ANALYTICAL ABSTRACTS

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Editor: NORMAN EVERS, B.Sc., Ph.D., F.R.I.C.
20, EASTCHEAP, LONDON, E.C.3
Telephone: MANsion House 6608

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1441. **Electrometric end-point detection in alkaline permanganate and hypochlorite titrations.** D. E. Woerner (*Dissert. Abstr.*, 1953, **13** [6], 976).—Electrometric methods cannot be used for the detection of the end-point of an alkaline permanganate titration. Amperometric titrations, with hypobromite prepared *in situ*, were devised for arsenite, ammonia and thiocyanate. These three systems have also been titrated on a semi-micro scale at concn. of the order of 10^{-4} *M* with an error of < 2 per cent. A. JOBLING

1442. **Theoretical treatment of the spectrophotometric titration of divalent cations with complexone III and metal-specific indicators.** J. M. H. Fortuin, P. Karsten and H. L. Kies (*Anal. Chim. Acta*, 1954, **10** [4], 356–372).—It is shown that the following are the optimum conditions, in order of importance, for obtaining a sharp end-point in the titration of a divalent metal with complexone III and with murexide as a metal-complexing indicator. The complex-forming capacity of the titrant must be $< 10^4$ times that of the indicator. This must be high and is attained by working at high pH values. The concn. of indicator must be as low, and that of the metal as high as possible. W. C. JOHNSON

1443. **Some recent applications of coulometry.** R. Gauguin (*Chim. Anal.*, 1954, **36** [4], 92–97).—The advantages of the use of coulometry at const. intensity of current in conjunction with conductimetric, spectrophotometric and amperometric methods are discussed, and examples of each of these types of usage are described. The source and magnitude of the experimental errors to be expected are also indicated. H. F. W. KIRKPATRICK

1444. **The determination of the number of electrons involved in irreversible reductions by microcoulometry.** G. F. Reynolds and H. I. Shalgosky (*Anal. Chim. Acta*, 1954, **10** [4], 386–399).—The number of electrons (n) involved in an electrolytic reduction is given by the equation $n = MX/FW$, where M = mol. wt. of the reacting substance, F = the Faraday, X = coulombs passed during the electrolysis and W = wt. (in grams) of the substance that has reacted. Three methods are used for evaluating W and X. W is derived from $W = (i_o - i_t)VC/i_o$, where i_o and i_t are the initial and final polarographic diffusion currents, V = vol. of solution electrolysed and C = the initial concn. of the reacting substance. X = average current × time and $(i_o + i_k)/2$ may be taken as sufficiently close for an approximation to the average current. This is the basis of the Arithmetic Average Method in which it is only necessary to determine a pair of i values over a known time interval; the results can be duplicated by measuring a number of such pairs. In the Integration Method, a continuous record of the current flowing during electrolysis is made and X is obtained by measuring the area under the current - time curve measured at constant potential. W is measured as before. The Graphical Method relies on the slope of a graph of log i against t being proportional to n. In each method the electrolysis is carried out at the dropping-mercury electrode (*cf.* Gilbert and Rideal, *Trans. Faraday Soc.*, 1951, **47**, 396). The construction of the polarographic cell is described, as well as the features of a polarograph particularly suited to the Integration Method. The Integration Method is the most accurate of the three, but, when a suitable polarograph is not available, the Arithmetic Average Method will serve, with certain reservations. The most desirable conditions of electrolysis are enumerated. W. C. JOHNSON

1445. **A study of some sources of error in the measurement of oxidation - reduction potentials. I.** E. S. Boatman (*J. Med. Lab. Technol.*, 1953, **11** [4], 224–240).—An apparatus is proposed for the determination of bacterial oxidation - reduction potentials. Wembley X8 glass was found to be suitable for the metal-to-glass seals, when 20 S.W.G. wire was used. Platinum electrodes fused directly into the walls of the culture tubes are unreliable. Several disadvantages, including the risk of contamination, were met in bubbling air or oxygen through the culture fluids, so the apparatus was modified to allow aeration of the cultures by shaking. When 15 different Pt - Hg electrodes were used to measure the E_H of horse digest broth (pH 7·6 at 37°C), the values ranged from 200 to 450 mV. After the same electrodes had been dry heated at 160° C for 2 hr., the variation was much less, *viz.*, 220 to 300 mV, although still too great. F. W. DIGGINS

1446. **A study of some sources of error in the measurement of oxidation - reduction potentials. II.** E. S. Boatman (*J. Med. Lab. Technol.*, 1954, **12** [1], 23–39).—Electrodes were made as nearly identical as possible and were tested in standard cells. Hydrogen peroxide gave a cell of high E_H and Na thioglycollate one of medium E_H; for low E_H, 0·1 per cent. agar in 1 per cent. peptone - water medium at pH 7·6 containing 0·25 per cent. of Na thioglycollate was used. Apparently identical electrodes gave different readings in the standard cells, but agreement was better after standing them in conc. nitric acid for 15 min. and then washing and dry heating them at 160° C for 2 hr. After an initial high reading, they came to a lower steady level in about 40 min. The differences between electrodes were then of the order of 10 to 20 mV. When readings were taken at 5-min. intervals with an electrode continuously immersed in the system, the readings were nearly identical over a period of 1 hr. If, however, the electrode was removed, washed, stored in distilled water for 5 min., then dried and replaced in the same system, the readings were erratic. Electrodes taken from high to low E_H and *vice versa* did not give the same readings as control electrodes already in the systems, even when the test electrodes were

left in the systems for 2 hr. Electrodes that gave consistent readings when tested against a quinhydrone half-cell gave widely different values with biological systems. Saturated KCl - agar bridges of bore diameter ranging from 1 to 10 mm gave no improvement in readings. A bore diameter of 1 mm is recommended. F. W. DIGGINS

1447. **Formation of analytical precipitates.** J. D. O'Rourke (*Dissert. Abstr.*, 1953, **13** [6], 975–976).—Nucleation and crystal growth in the pptn. of $BaSO_4$ are studied by nephelometric and conductimetric methods. The results provide an explanation of the observed induction period; theoretical equations are derived. A low activation energy suggests that the nuclei arise from simple ionic association. The dependence of the rate of nucleation on [activity]^4 suggests that the nucleus is a quadripole. A. JOBLING

1448. **Principles and techniques of chromatography.** R. J. Dimler (*Trans. Amer. Ass. Cereal Chem.*, 1954, **12** [1], 1–28).—A survey with 36 references of the development of partition, adsorption and ion-exchange chromatographic methods of separation and an outline of variations in technique and some of the practical applications of the method are given. M. TADMAN

1449. **The relation between concentration and total optical density in radial inorganic paper chromatography.** C. Bergamini and W. Versorese (*Anal. Chim. Acta*, 1954, **10** [4], 328–334).—Radial paper chromatograms are prepared by the method of Berlingozzi and Serchi (*Brit. Abstr. C*, 1952, 580), definition being improved by use of a complex-forming solvent mixture of 0·5 g of benzoylacetone, 50 ml of butanol and 50 ml of 0·1 *N* HNO_3. The chromatograms are sprayed with appropriate reagents. R_F values are as follows: Pb 0·16, Ag 0·24, Cu^{II} 0·32, Cd 0·21, Co 0·20, Ni 0·20, Tl^{I} 0·18. The optical density of each band is measured at 1-mm intervals along two orthogonal diameters and when these values are plotted the figure obtained is a triangle the base of which is the horizontal axis of the graph. The total optical density, represented by the area of the triangle, is proportional to the original concn. for any one cation. W. C. JOHNSON

1450. **Conductimetric detection of ions in paper chromatograms.** G. de Vries (*Nature*, 1954, **173**, 735–736).—Chromatograms of Li, Na and K were prepared with aq. soln. of their chlorides and perchlorates and a mobile ascending phase of pentanol - methanol (3 + 7 by vol.). The dried paper was passed at constant speed between two small steel cylinders having a const. p.d. of 4 to 80 V (d.c.) and the current density was read at intervals of 3 sec. (≡0·75 mm of chromatogram) on a spot galvanometer (coil resistance 450 ohms). The position of the alkali-metal ions was revealed by increases in conductivity. Each of the ions can be detected in the presence of the other two by this method, although a small amount of K may be masked by a large amount of Na. The limits of sensitivity are 0·5 μg for Li, 1 or 2 μg for Na and 3 or 4 μg for K. In presence of 500 μg of Na, 0·5 μg of Li was detectable. Perchlorates show satisfactory separation, but the method is less sensitive for these than for the chlorides. H. F. W. KIRKPATRICK

1451. **Quantitative evaluation of paper chromatograms by infra-red absorption.** J. D. S. Goulden (*Nature*, 1954, **173**, 646).—A solution of mixed amino-acids is spread across a strip of Whatman No. 50 filter-paper and developed for several hr. with 50 per cent. aq. ethanol; the solvent is evaporated and the strip is wetted with liquid paraffin to reduce reflection. An image of a Nernst filament is projected on the strip, and the central portion of the illuminated area is re-imaged on the entrance slit of an infra-red spectrometer; the paper is then drawn across the filament-image at a rate of 1 in. per min. The optical density at the max. of absorption is then plotted automatically against the distance traversed along the strip. For bands developed for approximately the same time, the optical density max. gives a smooth curve when plotted against the weight of material on the paper; the product of optical density max. and half-value width of the band has a nearly linear dependence on weight of material. Estimations may be made to ±5 per cent. by this method with three replicates. An example is given of the record for a mixture of glycine and alanine. H. P. PAGET

1452. **Paper chromatography of organo-metallic complexes: dithizonates.** G. Venturello and A. M. Ghe (*Anal. Chim. Acta*, 1954, **10** [4], 335–345).—The chromatographic behaviour of the dithizonates of Zn, Cd, Hg, Bi, Pb, Cu and Ag has been studied by means of filter-papers treated with buffer solutions of pH 1 to 10 and a series of alcohols, from methanol to *iso*pentanol, as developing solvents. From the results found with individual metals, conditions are evolved for the separation of certain binary and ternary mixtures. W. C. JOHNSON

1453. **Artificial contamination of chromatographic cells. Qualitative and quantitative effects.** A. Lacourt, G. Sommereyns and G. Wantier (*Mikrochim. Acta*, 1954, [2], 240–257).—Artificial contamination of chromatographic cells alters the dimensions of spots and their migration velocities and can be used to improve the evaluation of chromatographic spots, particularly with chromate and tungstate. The error can be reduced from 20 to 3 per cent. in 10-μg samples by the methods described. A. J. MEE

1454. **Factors influencing the applications of flame photometry.** W. G. Schrenk (*Trans. Amer. Ass. Cereal Chem.*, 1954, **12** [1], 64–71).—Methods for elimination or minimisation of interferences likely to cause error include improvements in instruments and use of internal standard technique, of standard soln. containing ions present in the unknown soln., and of buffers to render unimportant small variations in concn. of interfering substances and operational cleanliness. Data are given for min. concn. of Mn, Cu, Co, B and Fe determinable within the standard deviation of ±10 per cent. M. TADMAN

1455. **Use of ascorbic acid in industrial analysis.** D. L. Erleu (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 356–364).—Analytical uses of ascorbic acid are discussed with particular reference to determination of $Fe^{\cdots}$, ClO_3', IO_3', BrO_3' and $V^{\cdots\cdots}$, and to microcolorimetric determination of phosphates, germanates, silicates and arsenates. G. S. SMITH

1456. **New amides of thioglycollic acid and their analytical applications. VI. 4-Nitro-2-thioacetamidophenol. Qualitative analytical applications. VII. Quantitative applications.** F. Buscaróns and J. Artigas (*An. Soc. Esp. Fís. Quím., B*, 1953, **49** [5], 375–378; 379–386).—4-Nitro-2-thioacetamidophenol gives precipitates with Ag, Hg^{I}, Hg^{II}, Cu, Pd, As^{III}, As^{V}, Au, Pt and Se salts at pH 1 to 2. At

pH 2, Bi^{III} is pptd., and at pH 3, Pb^{II} and Cd are pptd. The reagent precipitates Co^{II} and Ni^{II} in presence of NH_3, and Tl^{I} in presence of KOH - KCN. The Ag, Hg, Cu and Cd salts are insol. and suitable for qualitative recognition of the metal ions. The monohydrated Ag and Hg^{II} salts can be used gravimetrically if pptd. at pH 1 to 2 and 80° to 90° C. By titration of the ppt. with I, the reagent can be applied to volumetric determination of Cd, Cu, Ag and Hg. D. P. YOUNG

1457. **5-Fluoro-8-hydroxyquinoline (5-fluoroxine) and its sensitivity towards certain metals.** R. G. W. Hollingshead (*Chem. & Ind.*, 1954, [12], 344–346).—5-Fluoro-8-hydroxyquinoline is prepared by coupling 8-hydroxyquinoline with diazotised sulphanilic acid, reducing the product with Sn^{++} to give 5-amino-8-hydroxyquinoline (yield 90·3 per cent.), treating this with aq. fluoroboric acid and pyrolysing the resulting compound to 5-fluoro-8-hydroxyquinoline (yield 39·2 per cent.). The sensitivity of the reagent (0·02*M* in ethanol) towards Cu^{++}, Al^{+++}, UO_2^{++}, Co^{++}, Mg^{++} and Hg^{++} at pH 5·3, 8·35 and 13·1 is discussed. The results are closely similar to those obtained with 8-hydroxyquinoline. D. A. PANTONY

2.—INORGANIC ANALYSIS

1458. **A rapid volumetric method for the determination of micro-amounts of sodium and potassium.** H. Flaschka and A. M. Amin (*Chemist Analyst*, 1953, **42** [4], 78–80).—Two rapid volumetric methods are described for the determination of micro-amounts of Na and K. In the first method the alkali sulphates are ignited and the residue is dissolved and made up to 10 or 20 ml. An aliquot of this solution is run, with washing, through a cation exchanger (illustrated), and the eluate is heated to boiling and titrated with 0·01 *N* NaOH, giving a value for the total alkali metals. A second aliquot is adjusted to pH 4 with acetic acid, 2 to 3 mg of NH_4Cl are added and the solution is heated to 70° C. Potassium is pptd. as $KB(C_6H_5)_4$ by adding $NaB(C_6H_5)_4$ and, after cooling, the ppt. is filtered, washed twice with 0·5 ml of H_2O, then with a wash solution [saturated soln. of $KB(C_6H_5)_4$] and finally with 2 or 3 drops of H_2O. The ppt. is dissolved in acetone and the soln. is evaporated and ignited in a platinum crucible, when the $NH_4B(C_6H_5)_4$ is volatilised and the K salt is converted to potassium metaborate, which is dissolved in a measured excess of 0·01 *N* HCl, boiled and titrated with 0·01 *N* NaOH, giving a value for the K in the mixture. In the second method, which is used when only a small amount of alkali is present, the residue of alkali sulphates, after ignition in a platinum dish, is dissolved in a few drops of H_2O, and 100 to 200 mg of boric acid are added. The mixture is evaporated to dryness and heated (gently at first) for 5 min. to convert the alkalis to metaborates. After cooling, the residue is dissolved in a measured excess of 0·01 *N* HCl, boiled and titrated with 0·01 *N* NaOH. When the titration is complete, the pH is adjusted to 4 with acetic acid and the K is determined as before. D. BAILEY

1459. **Entrainment of potassium by sodium nitrate crystallising from solution. Radiochemical estimation of sodium in potassium nitrate.** J. Pauly (*Compt. Rend.*, 1954, **238** [1], 80–82).—When $NaNO_3$ crystallises from aq. solution, KNO_3 (as impurity) is entrained according to the fractionation coeff. $k = 0·019$. The radiochemical estimation of $\lt 0·0001$ per cent. of $NaNO_3$ in KNO_3 depends on the different amounts of entrained ^{24}Na and ^{42}K ($k_{NaNO_3}/k_{KNO_3} \simeq 50$) formed by irradiation of the sample with thermal neutrons. $NaNO_3$ contents are extrapolated from a standard linear curve (sp. activity plotted against mg of $NaNO_3$) calculated from radioactivities of successive layers of crystals formed by cooling an irradiated mixture of KNO_3 (350 mg) and $NaNO_3$ (1·25 to 2·9 mg) immersed in saturated aq. $NaNO_3$ at 30° C. The error is $\simeq 0·02$ per cent. W. J. BAKER

1460. **Determination of sodium carbonate in sodium sulphide.** K. E. Stumpf (*Z. anal. Chem.*, 1954, **141** [3],190–197).—Aliquot portions of a soln. of commercial Na_2S are taken, and the CO_3'', SO_3'' and SO_4'' present are pptd. with an excess of 10 per cent. $BaCl_2$ soln. After 2 min. the ppt. is filtered off and washed first with water and then with 1 per cent. $BaCl_2$ soln. A measured excess of 0·5 *N* HCl and 0·1 *N* I soln. are added, when SO_3'' is oxidised to SO_4'', liberating an equiv. amount of acid at the same time as the $BaCO_3$ is dissolved. The excess of I is estimated with 0·1 *N* $Na_2S_2O_3$ soln. and the excess of acid with 0·5 *N* NaOH soln. The carbonate then corresponds to the sum of the acid originally added and that liberated when the SO_3'' is oxidised to SO_4'', less the alkali required for the back-titration. A correction has to be applied for the CO_2 taken up from the air after pptn. with the excess of $BaCl_2$, and a blank is evaluated from the results of 2 parallel estimations made with different aliquots of the sample soln. The value of the blank may be high, reaching 50 per cent. of the CO_3'' content on a 2-g sample containing 1 per cent. of Na_2CO_3, whilst the absolute error is $\pm 0·1$ to 0·2 per cent. The accuracy is generally sufficient, and represents a considerable improvement on the results given by other methods. J. H. WATON

1461. [**Determination of potassium by precipitation as potassium tetraphenyl boron.**] A. J. Nutten (*Ind. Chem.*, 1954, **30**, 57–59).—Recent work on the determination of K by pptn. with $NaB(C_6H_5)_4$ and its application to the determination of K in blood serum (*cf. Anal. Abstr.*, 1954, **1**, 20) and of Rb, Cs, choline, and so on (*cf. Brit. Abstr. C*, 1953, 213 and 497) are reviewed. A. R. PEARSON

1462. **The photometric determination of copper as sulphate.** W. Nielsch and G. Böltz (*Z. anal. Chem.*, 1954, **141** [5], 321–326).—Copper compounds have an absorption max. above 750 mμ and both nitrate and sulphate soln. can be estimated directly in an ELKO II photometer with the filter S75E. For soln. containing 200 mg of Cu with 4 to 8 ml of HNO_3 in 100 ml, the presence of conc. H_2SO_4 in amounts between 10 and 20 ml does not effect the extinction. Up to 4 ml of HF have no effect if H_2SO_4 is also present; HF can therefore be used in the prep. of soln. of alloys containing Si. In the absence of H_2SO_4, HF interferes; H_3PO_4 and $HClO_4$ interfere even when H_2SO_4 is present. The method gives highly reproducible results, the error in the estimation of a sample of electrolytic Cu being as low as 0·0003 for a mean extinction of 0·4706. E. HAYES

1463. **Potentiometric titration with the ferri[cyanide] - ferrocyanide electrode. Determination of copper.** I. L. Teodorovich and I. K. Leushina (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 340–345).—The Cu^{II} soln. is treated with aq. NH_3 until the

$Cu(OH)_2$ ppt. just disappears, a few drops of 0·5 *M* $K_3Fe(CN)_6$ are added and the soln. is titrated during intermittent stirring with 0·2 *M* $K_4Fe(CN)_6$; a platinum indicator electrode and a saturated calomel reference electrode with a galvanometer as a null instrument are used. As^V does not interfere, and the determination of Cu in Paris Green can be carried out successfully. G. S. SMITH

1464. **The application of thio salts in analysis. II. Estimations based on decomposition of thio salts. Part C. Estimation of gold, platinum and antimony.** I. K. Taimni and R. P. Agarwal (*Anal. Chim. Acta*, 1954, **10** [4], 312–316).—Gold is determined in a soln. of its chloride by adding $\simeq$20 ml of 2 *N* Na_2S for every 50 mg of Au and boiling with an excess of 2 *N* HCl to ppt. Au_2S_3, which is washed with hot water, CS_2, ethanol and ether, dried in a desiccator and weighed, or washed with hot water, dried for 1 hr. at 105° to 110° C and weighed. Antimony is pptd. by the same procedure from a soln. of Sb K tartrate and the Sb_2S_3 is washed with hot water and dried at 105° to 110° C. Platinum is pptd. similarly by means of 25 ml of 2 *N* Na_2S for every 50 mg of Pt, but the final concn. of HCl is $\leqslant$6 *N*. The platinum sulphide is ignited and weighed as Pt. W. C. JOHNSON

1465. **Determination of magnesium by means of thiazole yellow.** S. Samson (*Chem. Weekbl.*, 1954, **50** [12], 213–218).—The method of Young and Gill (*Brit. Abstr. C*, 1951, 470) is rendered inaccurate by the irregular action of the salts in the compensating soln. on the stabilising action of the polyvinyl alcohol, but results are satisfactory with the use of a 1:2:1:2:4 (by vol.) mixture of aq. 0·03 per cent. thiazole yellow, 2 per cent. $NH_2OH.HCl$, 1 per cent. *p*-methylaminophenol, 2 per cent. polyvinyl alcohol and 10 *N* NaOH; the aq. NaOH is added finally with special care in order to obtain a clear soln. To 10 ml of the soln. containing 10 to 100 μg of MgO, are added 10 ml of the above reagent and 2 ml of the compensating soln. described by Young and Gill. The absorption is read on a photoelectric colorimeter after 25 min., the colour being stable for >1 hr. A calibration graph is given. P. S. ARUP

1466. **Increased colour stability in the determination of magnesium.** C. H. Yien and L. Chesnin (*Proc. Soil Sci. Soc. Amer.*, 1953, **17**, 240–242).—A titan yellow and two thiazole yellow procedures for determining Mg in soil and plant extracts are described. The thiazole yellow procedures cover a wider range of Mg concn. than does the titan yellow procedure, and one of them is suitable for determining Mg directly in the ammonium acetate extracts of soils. Results were very similar to those obtained by A.O.A.C. methods. J. Sci. Food Agric. Abstr.

1467. **Some qualitative reactions of zinc and ferricyanides.** F. Sierra and E. Monllor (*An. Soc. Esp. Fís. Quím., B*, 1954, **50** [1], 53–58).—Zinc ion is detected at dilution of 10 p.p.m. by the coloured ppt. obtained on adding to a mixture of $K_3Fe(CN)_6$, dil. H_2SO_4 and one of the bases *o*-toluidine, *o*-dianisidine, 1-naphthylamine or dimethyl-*p*-phenylenediamine, the reaction depending on the lowered oxidation potential of the ferri complex by the reduction of pH. Colorimetric detection of the $Fe(CN)_6'''$ ion at dilutions of 1 to 100 p.p.m. by mixture with a base of the above type and a zinc salt is also described. M. TADMAN

1468. **Rapid EDTA titration of zinc following thiocyanate extraction.** J. Kinnunen and B. Wennerstrand (*Chemist Analyst*, 1953, **42** [4], 80–83).—Zinc is determined by extracting the thiocyanate complex from aq. solution with *iso*butyl methyl ketone (methyl *iso*propyl ketone, *n*-butyl methyl ketone, 2-butyl phosphate and pentanol-ether are equally efficient), extracting the Zn from the organic solvent with NH_3 and titrating with standard disodium ethylenediaminetetra-acetate (**I**) soln. The most efficient extraction of the thiocyanate complex, which is formed by using an excess of NH_4CNS corresponding to at least 4 per cent. in soln., is obtained when the free acid is less than 5 ml of conc. HCl in 100 ml of soln. Interference by Fe is prevented by complexing with fluoride, whilst Cu and Ag are masked conveniently by the addition of thiourea. Ca and Pb interfere if fluoride is present, so when these metals are present in large amounts, the use of sodium pyrophosphate or tartaric acid is preferable. For the determination of Zn in a Zn concentrate, 2·5 g of the sample are decomposed with 5 ml of H_2O and 2 to 3 ml of Br, treated with 50 ml of conc. HNO_3 and boiled. The soln. is filtered, the residue is fused with potassium pyrosulphate, cooled, dissolved in H_2O and filtered, and the filtrates and washings are combined and diluted to 250 ml. An aliquot (25 ml) is neutralised with aq. NH_3, treated with 1 to 2 g of ammonium difluoride, 2 to 5 ml of saturated thiourea soln. and 30 ml of NH_4CNS soln., containing 500 g of NH_4CNS per litre, and extracted with 40 ml of *iso*butyl methyl ketone. The extract is treated with 30 ml of a buffer soln. containing 350 ml of conc. aq NH_3 and 54 g of NH_4Cl per litre of soln., and 50 to 100 ml of acetone and diluted to 400 ml with H_2O. After adding 2·5 ml of 20 per cent. aq. KCN and 10 drops of Eriochrome black T indicator, the soln. is titrated with 0·05 *M* **I** solution. At the end of this titration 4 per cent. formaldehyde soln. is added in small amounts and titration is continued until further additions of formaldehyde have no effect on the end-point. A similar but somewhat simpler procedure with a mixture of pentanol and ether (1 + 5) as the extracting solvent is used for the determination of Zn in $NiSO_4$. Both methods are rapid and accurate. Cd interferes in the determination, but small amounts can be neglected. D. BAILEY

1469. **Adipic acid: a selective reagent for the determination of mercury.** D. R. Idler (*Chemist Analyst*, 1954, **43** [1], 9–10).—The use of adipic acid as a precipitant for various metals is discussed, and a method is proposed for the determination of Hg. An aliquot of the Hg soln. containing 0·08 to 0·10 g of Hg is evaporated to dryness with HNO_3. The pH of the soln. of $Hg(NO_3)_2$ is adjusted to approx. 3 with 10 per cent. NaOH soln., and 35 ml of 0·03 *M* adipic acid soln. are added. The pH is adjusted to 2·5 and the ppt. is filtered and washed with water. The ppt. is extracted with 3 or 4 20-ml portions of sat. aq. NaCl. H_2S is passed into the extract and the soln. is boiled for 10 min. The adipic acid formed is titrated with 0·025 *N* NaOH. The Hg can also be determined gravimetrically, if suitable precautions are taken. The ppt. obtained as above is collected on a sintered-glass crucible, dried at 140° C and weighed. The ppt. should be completely soluble in sat. aq. NaCl. G. B. THACKRAY

1470. **Colorimetric determination of boron with sodium alizarinsulphonate.** R. Fernández Cellini and F. Alvarez González (*An. Soc. Esp. Fís. Quím., B*,

1954, **50** [1], 59–70).—The formation of the boron complex with sodium alizarinsulphonate has been studied with respect to time of formation (about 20 hr.), effect of temp. on colour (production accelerated, but colour less stable) and influence of light. Variations in concn. of acid and of sodium alizarinsulphonate and interference caused by nitrates and organic matter were also examined. The reaction is highly sensitive, differences of concn. of 0·01 μg of boron per ml being detectable. M. TADMAN

1471. **A highly selective procedure for the spectrophotometric determination of aluminium with 8-hydroxyquinoline and its application to the determination of aluminium in iron and steel.** A. Claassen, L. Bastings and J. Visser (*Anal. Chim. Acta*, 1954, **10** [4], 373–385).—Aluminium is commonly determined by extracting its 8-hydroxyquinoline complex from alkaline soln. with $CHCl_3$ and measuring the colour intensity of the extract (*e.g.*, Gentry and Sherrington, *Brit. Abstr. C*, 1946, 234). The use of KCN in existing methods prevents interference by Fe, Cu, Ni, Co, Ag, Cd and Zn, but no satisfactory methods are available for dealing with interference, particularly from V and Ti. The following modifications are therefore proposed. Both KCN and Na ethylenediaminetetra-acetate are added to make the first extraction at pH 8·5 to 9·5 more selective. A second extraction with $CHCl_3$ from a soln. adjusted to pH 5 leaves Be in the aqueous phase. A third extraction from a soln. first treated with H_2O_2 while acid and then made weakly ammoniacal leaves Ti, V, Ta and Nb in the aqueous phase. Bi, Ga, Sb and Zr are removed by addition of cupferron to a soln. 2 *N* in HCl and by extraction with $CHCl_3$. Indium is removed by addition of Na diethyldithiocarbamate and extraction with $CHCl_3$, the 8-hydroxyquinoline present being first removed by brominating and then extracting the dibromohydroxyquinoline. The procedure is varied to suit various conditions. Permissible limits are stated for Th, Sc, NO_3', F′ and PO_4'''. The methods have been applied to the determination of Al in standard samples of iron, steel, glass, phosphor bronze, die casting alloy and dolomite. W. C. JOHNSON

1472. **Tentative method for spectrochemical analysis of aluminium-base alloys by the point-to-plane spark technique.** Anon. (*A.S.T.M. Designation* E 101–53T. Issued 1953, 9 pp.).—This method, which is designed principally for the analysis of chill-cast discs, may be extended to cover the analysis of other forms of sample on which a flat surface at least $\frac{5}{8}$ in. wide can be machined. The spectrum is excited by a discharge between a pointed pure graphite electrode and the flat surface. Circuit parameters are given for various alternative excitation sources, including the Feussner-type, air-interrupter, rectified spark with rotary interrupter and normal condensed spark. Either a large or medium dispersion prism instrument may be used, or a grating spectrograph. Calibration is effected by use of a number of primary standards (obtainable from the National Bureau of Standards) supplemented by secondary standards available commercially in the U.S.A. Normal photographic and photometric procedures are used for interpreting the spectra on the basis of the internal-standard method. The elements that may be determined and the concentration ranges covered are: Si, 0·02 to 14·0; Cu, 0·001 to 10·0; Mg, 0·001 to 10·0; Zn, 0·03 to 8·0; Ni, 0·03 to 3·0; Fe, 0·02 to 2·0; Mn, 0·005 to 2·0; Pb, 0·03 to 0·7; B, 0·03 to 0·7; Cr, 0·01 to 0·5; Ti, 0·01 to 0·5; Sn, 0·01 to 0·5; Be, 0·0002 to 0·5; Ca, 0·0005 to 0·1; and Na, 0·0005 to 0·05 per cent. A detailed list is given of suitable analysis and internal-standard lines for the various elements to be determined, together with interfering spectral lines and the concn. of the responsible element at which interference is observed. Coefficients of variation range from 2 to 4 per cent. at concn. >0·5 per cent. B. S. COOPER

1473. **The influence of silica on the determination of aluminium by the 8-hydroxyquinoline method.** S. K. Dubrowo (*Silikat Tech.*, 1953, **4**, 311).—The accuracy of the 8-hydroxyquinoline method for determining Al has been investigated when as much as four times the amount (by weight) of SiO_2 as Al_2O_3 is present. Results are satisfactory when the SiO_2 is in molecular soln., but not if polymerised SiO_2 is present. With SiO_2 to Al_2O_3 ratios greater than 4 to 1, results are >10 per cent. too high and pretreatment with *N* NaOH soln. is recommended. B. J. W.

1474. **Chemical determination of aluminium on metallised paper.** P. P. Grad and A. J. Catotti (*Chemist Analyst*, 1954, **43** [1], 12–13).—A gasometric method of determining aluminium is given; it is suitable for estimating the thickness of aluminium films deposited on paper. The apparatus consists of a reaction flask, levelling bulbs and a graduated gas receiver; it can be made from ordinary laboratory equipment. The 10 per cent. aq. NaOH used is introduced into the reaction flask, replacing H_2O, the whole apparatus being free from air. An accuracy of 95 per cent. on approx. 3 mg of Al is claimed with an average deviation of $\pm 2{\cdot}3$ per cent. Improved accuracy and precision are attained with approx. 30 mg of Al. G. B. THACKRAY

1475. **Acetylacetone as an analytical extraction agent. Extraction of aluminium, gallium and indium.** J. F. Steinbach and H. Freiser (*Anal. Chem.*, 1954, **26** [2], 375–379).—The extraction characteristics of aluminium, gallium and indium are described. The acetylacetonates of these metals are white and are prepared by extraction of the acid soln. (pH 0·5) of the metal with acetylacetone to remove Fe and pptn. of the acetylacetonates by basifying the extracted water phase with *N* aq. NH_3. Recrystallisation from 95 per cent. ethanol gives colourless crystals for each. The solubility of acetylacetone in water, as a function of pH and ionic strength, is determined spectrophotometrically. This solubility is unchanged (17·0 g per 100 ml) between pH 6·5 and pH 1, but decreases gradually (probably because of the salting-out effect at the high acid concn.) below pH 1, whilst increase in ionic strength produces an appreciable decrease in solubility. Equilibrium extraction curves for aluminium, gallium and indium by acetylacetone are determined. The relation between the shape and position of these curves and the properties of the acetylacetone chelates is examined, and the usefulness of such curves in predicting the feasibility of analytical separations and determinations is indicated. Results show the possible separation of these metals by solvent extraction at different pH values. D. BAILEY

1476. **Polarographic determination of the onset of precipitation of metal hydroxides and the solubility products of aluminium and titanium hydroxides.** P. N. Kovalenko and V. N. Nestorovich

(*Ukr. Chem. J.*, 1952, **18** [6], 635–640).—The pH of first pptn. of $Ti(OH)_4$ and $Al(OH)_3$ is located by the sharp drop in the graph of the polarographic diffusion wave height plotted against pH. Values of $1{\cdot}61 \times 10^{-38}$ for the solubility product of $Ti(OH)_4$ and of $4{\cdot}8 \times 10^{-31}$ for that of $Al(OH)_3$ at 22° C are obtained. R. C. MURRAY

1477. **Fluorimetric determination of small quantities of gallium.** L. K. Bradacs, F. Feigl and F. Hecht (*Mikrochim. Acta*, 1954, [2], 269–276).—Traces of Ga in minerals, waters and meteorites can be determined by measuring the fluorescence intensity of the $CHCl_3$ soln. of the Ga - cupferron-morin complex. The ferric iron in a measured quantity of soln. is reduced by dropping the soln. through a silver reductor. HCl is added to make the liquid 6 *N*. The liquid is then further reduced by addition of about 8 ml of saturated $TiCl_3$ soln. The mixture is set aside overnight and extracted for 6 hr. with 250 ml of ether. The ether phase is separated, the ether is distilled off, and the residue is taken up with 3 *N* HCl. Some KH_2PO_2 is added for completion of reduction, the soln. is evaporated to dryness and the residue is dissolved in 2 ml of 3*N* HCl. Each 1 ml of liquid is treated with 1 ml of 2 *N* HCl and 2 ml of H_2O (two parallel determinations). Ten drops of cupferron and 2 drops of morin soln. are added. The test portions are then shaken for equal times. Six ml of $CHCl_3$ are added and the mixture is shaken. The fluorescence is then determined and compared with standards. One to six μg of Ga can be determined in 6 ml of $CHCl_3$. The accuracy is −6 to −8 per cent., so that if necessary a correction factor of 1·075 can be applied.
A. J. MEE

1478. **On the thermogravimetry of analytical precipitates. LXII. Determination of yttrium.** Clément Duval (*Anal. Chim. Acta*, 1954, **10** [4], 321–322).—The thermolysis curve of moist yttrium hydroxide shows that Y_2O_3 is obtained at $\simeq$856° C. $Y(OH)_3$ forms at $\simeq$365° C, but produces no horizontal section in the curve. The curve for Y oxalate shows an approx. composition $Y_2(C_2O_4)_3.2H_2O$ between 180° and 300° C and formation of Y_2O_3 at 680° C. The anhydrous oxalate, produced at $\simeq$ 375° C, gives rise to no horizontal portion.
W. C. JOHNSON

1479. **Volumetric determination of carbon and carbon dioxide.** J. Kinnunen and B. Merikanto (*Chemist Analyst*, 1954, **43** [1], 17–18).—A simple and inexpensive arrangement is described for the titrimetric determination of CO_2 evolved in the combustion process for carbon. The CO_2 is absorbed in $Ba(OH)_2$ soln. and titrated with potassium hydrogen phthalate. The error for carbon is $\simeq \pm 5$ per cent. and for CO_2 $\simeq$1 per cent.
G. B. THACKRAY

1480. **A new method of determining carbon disulphide.** H. Zimmer (*Z. anal. Chem.*, 1954, **141** [4], 272–275).—The method described is particularly suitable for determining small quantities of CS_2 in benzene. Few materials likely to be present interfere; thiophen, amines, active sulphur and resins do not interfere and phenol interferes only by its colour. Interfering colours may also be removed by distillation or fractionation. Strong oxidising and reducing agents and phenylhydrazine interfere but are not likely to be present. The method is based on conversion of CS_2 by strong KOH soln. to K xanthate, which then reacts with molybdate to give a red compound soluble in the benzene. Take 1 ml of sample expected to contain 0·04 to 0·10 per cent. of CS_2 and add 2 ml of 10 per cent. alcoholic KOH soln. After 15 min. add 0·2 ml of reagent soln. (20 g of anhydrous ammonium molybdate in 100 ml of conc. HCl, heat to dissolve, add 100 ml of H_2O and 2 ml of 30 per cent. H_2O_2 and boil to remove Cl; pH 1·5; stable indefinitely) and 2 ml of H_2O and shake. Compare the red benzene layer with freshly prepared standards.
P. S. STROSS

1481. **Characterisation of occupations endangered by carbon disulphide.** H. Demus (*Faserforsch. u. Textiltech.*, 1954, **5** [2], 65–67).—Two suitable methods for estimating the risks attendant upon work in CS_2-containing air are described. The first depends upon a simple colorimetric test of the change in colour of copper amine solutions, brown cupric diethyldithiocarbamate being produced. Inhaled CS_2 is mostly absorbed by the body, and the second test measures the CS_2 content of the urine of exposed persons. A table is given showing the results of a number of tests over a working week, together with a daily test of the CS_2 content of the air in the vicinity.
M. TADMAN

1482. **Rapid determination of silica in glass.** H. Flaschka and A. M. Amin (*Chemist Analyst*, 1954, **43** [1], 6–7).—A method of determination of silica in glass on a cation-exchange column is proposed. Approx. 0·15 g of sample (*W*) is ignited with HF and H_2SO_4 in a platinum crucible, and reweighed (*R*). The residue of oxides and sulphates is washed into a beaker and treated with Wofatit K (acid form) for 10 min. at 70° C. The soln. is then passed through a column (1·2 cm in diam. by 8 cm long) and the column is rinsed with 50 ml of distilled water. The eluate is heated to boiling and, after cooling, titrated with 0·1 *N* alkali (*V* ml) against methyl red - methylene blue mixed indicator.

$$SiO_2 \text{ per cent.} = 100\ (W - R + 4{\cdot}003\ V)/W.$$

The time reqd. is 2 hr. and the max. error is $\pm$0·5 per cent. of SiO_2. The method is not applicable when borates are present, and a correction must be made for sulphates initially present.
G. B. THACKRAY

1483. **State of silicic acid in solution and its colorimetric determination.** M. M. Piryutko and Yu. A. Shmidt (*Bull. Acad. Sci., U.S.S.R.*, 1953, [4], 607–614).—Decrease of pH causes dissolved SiO_2 to polymerise to a form in which it does not react with molybdate reagent, and so falsifies colorimetric determinations. Complete depolymerisation can be effected by adding alkali and heating, or by adding NaF to an acid soln. at room temp.; colorimetric determinations then give satisfactory results.
R. C. MURRAY

1484. **Conditions of separation and gravimetry of silica. I. Determination of silica in silicates.** E. N. Egorova (*Bull. Acad. Sci., U.S.S.R.*, 1953, [3], 419–428).—In spite of careful adjustment of the conditions of dehydration and washing of precipitated SiO_2, small quantities are always lost through the filter during washing, owing, it is suggested, to the completeness of insolubilisation, which depends also on the degree of polymerisation of the SiO_2 produced during treatment prior to dehydration.
R. C. MURRAY

1485. **Use of complexones in polarography. I. Determination of titanium.** S. I. Sinyakova (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 333–339).—The

reduction of the Ti complex of ethylenediaminetetra-acetic acid on the dropping-mercury electrode occurs reversibly. The half-wave potential with reference to the saturated calomel electrode depends on the pH, and is −0·555 volt at pH 5·5, −0·427 at pH 4·6, −0·418 at pH 3·4 and −0·391 at pH 2·25. At lower pH values the complex tends to be destroyed. The clearest waves are obtained in 2 *M* Na acetate and 0·1 *M* ethylenediaminetetra-acetic acid (**I**). The diffusion current const. in milliamp. per millimol. per litre for Ti concn. of 0·234 to 2·340 millimol. per litre is 3·17 ± 0·11; over this range of concn. linearity between current and concn. is observed. Addition of **I** must be made before that of Na acetate as otherwise Ti forms a complex with acetate that is only slowly destroyed by **I**. Fe gives a complex with half-wave potential in 2 *M* acetate of −0·12 volt and does not interfere unless its concn. is 3 or 4 times that of Ti. V and Cu if present must be removed. G. S. SMITH

1486. **Photometric determination of traces of germanium after carbon tetrachloride extraction.** W. A. Schneider, jun., and E. B. Sandell (*Mikrochim. Acta*, 1954, [2], 263–268).—The extraction of $GeCl_4$ (GeO_2 in HCl) by CCl_4 has been examined. Extraction from 8 to 9 *M* HCl is most advantageous. The extraction coeff. (50 to 500 approx.) is independent of the original concn. of Ge. For the low concn. of Ge encountered in trace analysis, equilibrium is attained rapidly after 1 to 2 min. of shaking. Phenylfluorone was the most suitable colorimetric reagent. It is more sensitive than the reduced haematoxylin reagent and does not require the use of a buffered solution. It gives no colour with SiO_2. Ga, Ti, Sn^{II}, As^{III}, As^{V}, Sb^{III}, Bi, Mo^{VI}, Fe^{III}, Nb, Ta, Zr and W^{VI} give coloured products, but of these only As^{III} is extracted in large amounts by CCl_4 from conc. HCl soln. The method can be applied to silicate rocks. Silicates can be decomposed with HF - HNO_3 - H_2SO_4 without loss of Ge. Small amounts of chloride (<0·05 per cent.) do not cause loss of Ge. The soln. to be extracted (20 to 25 ml) should contain 0·5 to 10 μg of Ge, and should be 8 to 9 *M* in HCl. Oxidising agents liberating Cl must be absent or prevented from interfering by addition of $FeSO_4$. The soln. is shaken for 2 min. with 10 to 15 ml of CCl_4 and then the extract is shaken with 6 ml of H_2O for 2 min. After separation of the phases, 5 ml of the aq. soln. are pipetted into a 10-ml flask, 1 ml of gum acacia (1 g in 200 ml of H_2O) and 3 ml of phenylfluorone solution are added, and the mixture is made up to the mark. After 1 hr. the transmittance of the soln. at 510 mμ is determined against a reagent blank. A standard graph is prepared. For silicate rocks, 0·5 g of the finely powdered sample is treated with H_2SO_4 - HNO_3 - HF and evaporated to fumes of H_2SO_4. Water is added and the evaporation is repeated twice. The soln., after addition of HCl, may then be dealt with as above. The lower limit with a 0·5-g sample is 0·1 to 0·2 p.p.m. A. J. MEE

1487. **Determination of germanium as 12-tungstogermanate.** L. H. Phifer (*Dissert. Abstr.*, 1953, **13** [6], 968).—A gravimetric method is described for the determination of germanium as tetraphenylarsonium 12-tungstogermanate by the reaction of germanate in the presence of excess of tungstate with $(C_6H_5)_4AsCl$. The ppt. is washed with dil. HNO_3, dried at 110° C, and weighed as $[(C_6H_5)_4As]_4GeW_{12}O_{40}$ (factor for Ge = 0·0163). For complete reaction, it is essential to use a paratungstate soln. to maintain the pH between 5 and 6 and the temp. between 95° and 100° C. Any excess of the paratungstate must be removed before addition of the $(C_6H_5)_4AsCl$. Other anions that form heteropoly acids and reducing anions must be removed. Citrate, tartrate, oxalate and acetate also interfere. A large group of anions that react directly with the $(C_6H_5)_4As^+$ must be absent. Cl^- must be absent to prevent loss of Ge as volatile $GeCl_4$. Accuracy is good when the quantity of Ge lies between 0·04 and 6 mg. An isotope dilution method for determining Ge is suggested. The solubilities of GeS_2 and Mg_2GeO_4 in solutions from which they are usually pptd. are $2{\cdot}2 \times 10^{-4}$ g-mol. per litre at 30° C and $1{\cdot}3 \times 10^{-5}$ g-mol. per litre at 29° C, respectively. The solubilities of the cinchonine and $(C_6H_5)_4As^+$ ppt. of 12-tungstogermanic acid are $<3{\cdot}9 \times 10^{-6}$ g-mol. per litre at 30° C. A. JOBLING

1488. **A simple volumetric method for the estimation of zirconium and its application to zircon concentrates and related materials.** P. R. Subbaraman and K. S. Rajan (*J. Sci. Ind. Res., B, India*, 1954, **13** [1], 31–34).—The Zr mineral is fused with Na_2O_2, the melt is extracted with water, evaporated to the fuming point with $HClO_4$ and an aliquot of this solution (ZrO_2 ≯ 100 mg) is passed through a Jones reductor. The pH is adjusted to 1·5 with dil. aq. NH_3, the soln. is warmed to 60° C, and an excess of standard KH_2PO_4 is added. The soln. is kept on a water-bath for 1 hr., then cooled to room temp., and the excess of KH_2PO_4 is titrated with bismuthyl perchlorate, with the aid of a soln. of *s*-di-(N-allylthiocarbamyl)hydrazine in $CHCl_3$ as indicator. ThO_2, in amounts up to 3 per cent. of the ZrO_2 content, does not interfere. Interference by Fe is eliminated by reduction in the Jones reductor, or by means of magnesium ribbon, before pptn. of the Zr. The method is as accurate as the usual gravimetric procedure. J. M. JACOBS

1489. **The conductimetric determination of small quantities of lead.** A. Schneider and H. Beisken (*Z. anal. Chem.*, 1954, **141** [5], 326–336).—Small amounts of lead (0·2 to 0·005 mg) are determined by conductimetric titration with H_2S water. From experimental studies the main sources of error were determined. Changes in the titre of the H_2S soln. are due either to variations in the relative volumes of the liquid and gas in the containing vessel or to temp. changes. Conductivity values change with temp. at the rate of 2 per cent. per degree. Impurities or CO_2 in the distilled water used can cause errors of as much as 10 per cent. In neutral soln. of $Pb(NO_3)_2$ (containing 0·1 mg of Pb) the curve shows an anomalous min. at the beginning of the titration; this is due to adsorption of H ions and can be avoided by adding HNO_3 to make the soln. 10^{-3} *N*. The total error can be reduced to ±2 per cent. for 0·02 mg of Pb, and to ±10 per cent. for 0·005 mg. E. HAYES

1490. **Thorium determination in deep-sea sediments.** E. Picciotto and S. Wilgain (*Nature*, 1954, **173**, 632–633).—A combination of chemical and photographic methods has been shown previously (*Nature*, 1953, **171**, 742) to permit individual determinations of the activities of the Th isotopes of mass numbers 227, 228, 230 and 232. Methods of correcting for the interference by ^{227}Th in the determination of ^{228}Th are considered. The concn. of Th in samples of red clay from the central Pacific Ocean was determined; the mean value is ≃ 5 p.p.m., considerably higher than that reported for clay from the Atlantic Ocean. The difference may however be

due to incomplete extraction of Th from the latter. Previous methods for dating deep-sea sediments by determination of the ratio of Ra to ^{230}Th are open to objection; determination of the ratio of ^{230}Th to ^{232}Th should give a valid method, in which the assumptions made can be readily checked.

H. P. PAGET

1491. **The semi-micro Kjeldahl method for the determination of nitrogen.** J. K. Fawcett (*J. Med. Lab. Technol.*, 1954, **12** [1], 1–22).—For 0·2 ml of plasma, 1 ml of conc. H_2SO_4 and a digestion time of 20 min., various concn. of the following catalysts or digestion promoters have been investigated: K_2SO_4, $K_2S_2O_8$, HgO, Se, $Na_2SeO_4.10H_2O$, $CuSO_4.5H_2O$ and H_3PO_4. The NH_3 was distilled from a Markham still (*Biochem J.*, 1942, **36**, 790) into H_3BO_3 soln. containing methyl red and bromocresol green indicators. Selenium gave the highest percentage recovery with an optimum concn. of 100 mg. Sodium selenate was next best and HgO third. Digestion for 2 hr. in presence of Se plus much Na_2SO_4, or in presence of a large amount of Na_2SeO_4 caused loss of nitrogen. In absence of sulphate quite large amounts of Se had no detrimental effect. One ml of a digestion mixture made by boiling 10 g of Se in 100 ml of H_2SO_4 was added to the digestion flask to give the optimum concn. of Se. With this soln. a 30-min. digestion of plasma, urine or protein-free filtrate gave the same recovery as a 3-hr. digestion, but an 18-hr. digestion showed some loss of nitrogen from plasma. For gastric juice, faecal suspension or milk, 1 hr. was satisfactory. Digestion does not necessitate clearing the mixture (*cf.* Middleton and Stuckey, *J. Pharm. Pharmacol.*, 1951, **3**, 829). Extra acid was added when much fat or carbohydrate was present. Results with 26 and 22 per cent. of sodium sulphate for the pptn. of globulin from plasma in differential protein estimations showed no significant difference. Ether was used to separate the ppt. from the albumin - sodium sulphate soln. (*cf.* Kingsley, *J. Biol. Chem.*, 1940, **133**, 731; Sobel, Mayer and Gottfried, *Ibid.*, 1944, **156**, 355). Digestion (1 hr.) of 4 ml of this soln. was best with 1 ml of conc. H_2SO_4 and 5 mg of Se. For samples containing 0·2 to 10 mg of nitrogen, precision is ± 1 per cent.

F. W. DIGGINS

1492. **Analysis of gases in steel: nitrogen.** C. Artero Soteras (*Inf. Quim. Anal.*, 1954, **8** [1], 11–17).—Nitrogen in steel occurs in three possible forms: occluded as gas, dissolved in the iron as a solid solution, or combined as nitrides. The nitrogen content varies according to the method of smelting, being least in fusion *in vacuo* and most in the Bessemer process. Methods that have given excellent results include (*i*) distillation and volumetric estimation, (*ii*) distillation and colorimetric estimation and (*iii*) direct colorimetry. In the first, 3·5 g of sample are treated with 50 ml of dil. H_2SO_4(1 + 4); 5 g of K_2SO_4 are then added, the flask is fitted with an air condenser, the contents are gently warmed at first, and then the heating is increased. After this treatment, the mixture is cooled, diluted with 35 ml of water and washed into a special type of steam-distillation flask. Sodium hydrogen tartrate is added and the liberated ammonia is distilled into 10 ml of 0·1 per cent. boric acid. The excess of acid is titrated with 0·01 *N* HCl and methyl red - methylene blue indicator (0·125 g of methyl red and 0·083 g of methylene blue in 100 ml of pure ethanol).

When the steel sample contains less than 0·01 per cent. of N, the distillation can be made into Nessler's reagent and the colour produced can be matched with standard NH_3 soln.

In the third method the sample is dissolved in acid, heavy metals are pptd. with NaOH and filtered off, and the filtrate is tested directly with Nessler's reagent on a colorimeter. For samples containing 0·001 to 0·015 per cent. of N a 1-g sample is taken, for 0·015 to 0·03 per cent. 0·5 g, for 0·03 to 0·075 per cent. 0·2 g, and for greater percentages 0·1-g samples are used.

H. PRITCHARD

1493. **Studies with hydroxamic acid. III. Micro-detection of hydroxylamine as ferric hydroxamate. Spot test and micro-tube technique.** O. A. Guagnini and E. E. Vonesch (*Mikrochim. Acta*, 1954, [2], 211–212).—The detection of hydroxylamine as ferric hydroxamate can be carried out as a spot test or in a micro-tube. A soln. of 20 per cent. formaldehyde containing 0·5 g per litre of ferrous ammonium sulphate acidified with 0·5 ml of conc. H_2SO_4 per litre is used (solution A). For the tube test, one or two drops of soln. A and 10 mg of potassium persulphate are added to one drop of the test liquid. The mixture is boiled for 1 to 2 min. After 5 min. a pale rose colour develops with 0·2 μg of hydroxylamine. With larger quantities a violet colour is produced. For the spot test, the materials are warmed to 50° to 60° C or exposed to an infra-red lamp. The sensitivity is 0·1 μg of hydroxylamine and the limiting concn. is 1:50,000. A. J. MEE

1494. **Carbonate variant of the mass-spectrographic analysis of water oxygen.** A. V. Trofimov (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 353–355).—Isotopic analysis of oxygen in water is effected by preliminary exchange with CO_2 according to the method of Mills and Urey (*J. Amer. Chem. Soc.*, 1940, **62**, 1019) at 100°C, when complete exchange is much more rapid.

G. S. SMITH

1495. **Reychler's reaction as a test for basic peroxides.** E. B. Melardi (*Ann. Chim., Roma*, 1954, **44** [1], 20–27).—The quantitative nature of Reychler's reaction, *e.g.*, $2ClO_2 + Na_2O_2 \rightarrow 2NaClO_2 + O_2$, is reviewed; supplementary work on Be, Zn, Cd, Hg, Zr, Th and Ni peroxides is also described. Acidic peroxides, such as those of Th, Zr or U, do not react in this way, so distinguishing them from basic ones, which generally react quant.; occasionally the reaction is the best means of producing a chlorite. The existence of Be and rare-earth peroxides and non-existence of an Al peroxide are demonstrated.

R. C. MURRAY

1496. **The analytical characteristics of hydrogen peroxide.** C. Duval (*Chim. Anal.*, 1953, **35** [11], 265–273).—An extended survey is given of the reactions of H_2O_2 that have analytical application. Physical data included comprise absorption bands in the i.r. and u.v.; Beer's law is not valid in u.v. light. Raman spectra are given for H_2O_2, D_2O_2 and HDO, although they have no analytical use. Qual. tests include several spot tests; Denigès reagent is recommended as the best for use with the microscope. The spot tests are (*i*) reduction of Ni_2O_3, (*ii*) formation of Prussian blue by which 0·08 μg can be detected at a limit of dilution of 1 in 600,000, (*iii*) change of blue colour of perchromic acid to violet-red in ether on addition of diphenylcarbazide with a sensitivity of 5 μg in 5 to 10 ml, (*iv*) restoration of colour to phenolphthalein reduced by zinc in alkaline soln., (*v*) 12 organic colour

reagents and (*vi*) fluorescence of PbS paper in u.v. with a limit of 0·01 μg at a dilution of 1 in 5×10^7. No gravimetric method exists for quant. determination of H_2O_2. Volumetric methods discussed include determination in presence of S_2O_3'' and SO_3'' and alkaline oxalates. $Ce(SO_4)_2$ is recommended in the presence of organic materials, and allows the determination of H_2O_2 in the presence of aliphatic per-acids, which can then be determined by iodimetry. $Mn_2(SO_4)_3$ is preferred to $KMnO_4$. Polarographic determination is discussed, the reduction wave of O in H_2O_2 is reversible at all pH values, but the reduction wave of H_2O_2 is irreversible. Applications of polarographic technique include the determination of ether peroxide in the presence of H_2O_2. Colorimetrically H_2O_2 can be determined by (*i*) Ti salts, (*ii*) $KMnO_4$ catalysed by $MgSO_4$ (at 525 mμ), (*iii*) Mohr's salt with CNS′, (*iv*) decomposition or production of H_2O_2 by certain bacteria. Reactions giving O can be followed by gas volume and include those with MnO_4^-, $Fe(CN)_6'''$, HOBr, $CaOCl_2$, NaOCl or MnO_2. H_2O_2 can be estimated colorimetrically or used to estimate (*i*) $KMnO_4$ catalysed by MnO_2 (at 525 mμ), (*ii*) $Fe^{··}$ with CNS′ (at 550 mμ or 480 mμ for low concn.), preferably between pH 1 and 2, (*iii*) Ti^{IV} (at 410 mμ or with a blue filter), 0·1 g per ml can be estimated with an error < 3 per cent., or (*iv*) I′ catalysed by molybdate, free I being measured directly at 350 mμ, or with starch at 600 mμ. W. C. WAKE

1497. **The determination of sulphur by combustion.** M. Pontet and R. Boulin (*Chim. Anal.*, 1954, **36** [4], 98–101).—Sulphur in steels is determined by combustion of the sample in a current of oxygen, the gases produced being absorbed in $AgNO_3$ soln. (5 g per litre), and the acid produced is titrated with 0·01 *N* NaOH with the aid of a mixed indicator consisting of 0·1 per cent. alcoholic solutions of methylene blue and methyl red (2 + 1 by vol.). A controlled combustion temp. of 1400° C and oxygen flow rate of 1·5 litres per min. were found to be essential to good recovery. The method avoids the use of a correction factor and yields results in satisfactory agreement with gravimetric ones. H. F. W. KIRKPATRICK

1498. **The silver absorbent method for sulphur with milligram and decimilligram samples.** J. A. Kuck and E. C. Grim (*Mikrochim. Acta*, 1954, [2], 201–210).—A rapid method for the determination of S in org. compounds in the absence of halogens or alkali metals, based on the formation of Ag_2SO_4 and direct determination of the increase in weight, is described. A rapid flow of O is maintained (55 to 60 ml per min.) and pyrolysis of the sample takes place in a quartz capsule. The Ag is used in the form of a gauze roll, which is cleaned in dil. HNO_3(1 + 1), washed with water and ethanol, dried and ignited in O to const. wt. The procedure was used first with milligram samples. The standard deviation for four samples varied from $\pm$ 0·10 to $\pm$ 0·16. Twenty-six 2 to 3-mg samples (with and without P as an additional element) were analysed by traditional methods and by the Ag absorbent method. Most of the results by the two methods agreed to within 0·3 per cent. of S and one differed by more than 0·5 per cent of S. There was no evidence that the Ag absorbent method gave consistently high or low values. The procedure was extended to the analysis of decimilligram samples by the use of the Garner quartz-fibre balance; the lower limit of the method lies between 133 μg and 55 μg of S for these samples. Results are given of a block experiment in which the effect of two variables, size of sample and type of experiment was investigated. Statistical analysis of the data shows that the Garner quartz-fibre balance when used with decimilligram apparatus is superior to a good microchemical balance with normal equipment in the range 267 to 133 μg of S. A. J. MEE

1499. **The use of bromine water in the determination of sulphur in coal or coke by the Eschka process.** T. Battye, R. Johnson and H. C. Wilkinson (*J. Appl. Chem.*, 1954, **4** [3], 119–123).—Addition of Br water to oxidise sulphites in the sintered mass when using the Eschka method can be avoided if the incineration is effected by means of a Bunsen burner or in a ventilated muffle furnace (as recommended in B.S. 1016:1942). Temp. of 700° to 800° C are attained under completely oxidising conditions and, when 1 g of coal or coke is mixed with 4 g of anhydrous Na_2CO_3 and 8 g of light MgO, no sulphite is present in the sintered mass. W. J. BAKER

1500. **On the volumetric determination of hydrogen sulphide and soluble sulphides.** P. O. Bethge (*Anal. Chim. Acta*, 1954, **10** [4], 310–311).—Sulphide ($\simeq$ 2·5 mg) is determined by boiling it for 10 min. with an excess of KIO_3 in 4 *N* NaOH, acidifying, and titrating the excess of KIO_3 with 0·1 *N* $Na_2S_2O_3$. Potassium iodate is a more suitable oxidant than NaOCl or $KMnO_4$, both of which undergo some decomposition on boiling with strong NaOH (*Anal. Abstr.*, 1954, **1**, 72). W. C. JOHNSON

1501. **A sensitive electrometric method for the determination of sulphide ion in solution.** R. E. Press and K. A. Murray (*J.S. Afr. Chem. Inst.*, 1952, **5** [1], 45–54).—Iodine is electrically generated in solutions (pH $\simeq$ 6) containing sodium azide, KI, HCl and the sulphide-containing sample, the excess at the end of the reaction being detected amperometrically. Sulphide ion from 0·8 to 0·01 p.p.m. can be determined, and is detectable down to 0·0025 p.p.m. with 95 per cent. certainty. Graphs relating change of reactant concn. to time, reactant concn. to rate of change of iodine concn., and change of generation rate to time are given. M. TADMAN

1502. **Colour reaction of alkaline sulphites with Bougault's hypophosphite reagent in presence of sodium tungstate.** L. Blanchard (*Ann. Falsif.*, 1954, **47**, 31).—In presence of $\simeq$ 5 ml of aq. 1 per cent. Na tungstate and 0·5 ml of Bougault's reagent (conc. HCl soln. of NaH_2PO_2), alkaline sulphites or SO_2 give, on gentle heating, an intense sky-blue coloration accompanied, at high concn. of SO_3'', by colloidal S. The sensitivity of the test is $\simeq$ 10 mg of Na_2SO_3 per litre, and it can be used for the detection of sulphite adulterant in meat, the test being made on the filtered protein extract obtained by defecation with either H_2WO_4 or $Zn_2Fe(CN)_6$. W. J. BAKER

1503. **A new iodimetric method of analysis for sulphites or mixtures of sulphite, sulphate and alkali sulphides.** H. N. Terem and A. H. Isnel (*Rev. Fac. Sci. Univ. Istanbul*, 1953, **18A**, 250–259).—The direct titration of alkali sulphites with KI in a CO_2 atmosphere (after dissolving the solid sample in oxygen-free water in the same atmosphere) is found preferable to titration in air with anti-oxidants. The suggested method permits boiling and re-cooling of the soln. before analysis without

loss of SO_3''. For mixtures of alkali sulphates, sulphites and sulphides, the sample is dissolved as above, the H_2S then being driven off by gentle heating in a CO_2 stream that carries it over to be absorbed in KI in other vessels and thus to be determined with thiosulphate on cooling. The sulphite is determined as above, and sulphate is finally pptd. as $BaSO_4$ and corrected for the oxidised sulphite. CHEM. ABSTR.

1504. **Determinations of thiosulphate and nitrite.** R. H. Pierson (*Anal. Chem.*, 1954, **26** [2], 315–320).—A convenient procedure is devised for determining thiosulphate and nitrite in aq. ammoniacal soln. containing no S'' or SO_3''. On treatment with $AgNO_3$, under carefully controlled pH conditions, thiosulphate yields Ag_2S quantitatively and nitrite is unaffected. The S_2O_3'' determination is then completed by measuring the Ag content of the Ag_2S ppt. by dissolving it in HNO_3 and titrating with ammonium thiocyanate in accordance with the Volhard procedure; NO_2' is determined in the filtrate from the $AgNO_3$ treatment by ceric ammonium sulphate. Recoveries of 99 to 101 per cent. for both ingredients are consistently attained. An improved method described for determining thiosulphate in $(NH_4)_2S$ soln. containing no NO_2' or SO_3'' is applicable to soln. containing as little as 0·1 per cent. by wt. of thiosulphate ion (about 0·01 *M* in thiosulphate) and as much as 18 per cent. by wt. of S''. It is based on removal of the sulphide, polysulphide and hydrosulphide as H_2S and S by bubbling N through the buffered soln. Preliminary experiments indicate that the method could be extended to the analysis of Na_2S or K_2S solutions. From 99 to 101 per cent. thiosulphate and from 98 to 99·5 per cent. nitrite could be recovered from freshly prepared $(NH_4)_2S$ solutions by a procedure described, in which the N-purging is combined with the $AgNO_3$ separation. Qualitative procedures for detecting sulphite in the presence of thiosulphate and in the presence of both thiosulphate and nitrite, are described as applied to buffered soln. A red colour develops in 5 to 10 min. after adding 5 ml of selenious acid soln. and 5 ml of dil. HCl in the first test, and 5 ml of selenious acid soln., 2 g of sulphamic acid and 5 ml of dil. HCl in the second test. As little as 0·2 mg of SO_3' can be detected in the presence of as much as 100 mg of thiosulphate, and as little as 0·5 mg of SO_3' in the presence of as much as 100 mg of thiosulphate and 50 mg of nitrite. Chloride in moderately large amount will not interfere. O. M. WHITTON

1505. **Polarographic determination of sulphate ion in glacial acetic acid.** W. Kemula and A. Krzemińska (*Roczn. Chem.*, 1954, **28** [1], 125–133).—The method has been designed to determine small amounts of SO_4'' (up to $5{\cdot}66 \times 10^{-5}$ mole per litre) in pure glacial acetic acid. This is diluted with 3 times its weight of dist. water, and a known excess of standard $Pb(NO_3)_2$ soln. ($Pb'' = 1{\cdot}35 \times 10^{-5}$ mole per litre) is added. In order to render the $PbSO_4$ ppt. sufficiently insol., 96 per cent. ethanol is added until its concn. is 50 per cent. The Pb remaining in soln. is determined polarographically by the aid of specially designed calibration curves. The method is suitable for routine tests in the production of pure glacial acetic acid; its accuracy is $\pm 0{\cdot}7 \times 10^{-5}$ mole of SO_4'' per litre of acetic acid. A Heyrovský polarograph was used in the investigation, and the pure glacial acetic acid reagents examined were B.D.H. "AnalaR" and Merck products. The SO_4'' contents established were 0·00025 per cent. per litre for the first and 0·0004 per cent. per litre for the second; the guaranteed max. content was 0·0004 per cent. per litre for both. H. BURSTIN

1506. **Detection and volumetric determination of the tetrathionate ion alone or in mixtures.** M. Jacquinot (*Chim. Anal.*, 1953, **35** [11], 277–280).—A brief review of the reactions of S_4O_6'' is given followed by a detailed study of the colour reaction with $SbCl_3$ to form Sb_2S_3. Ions studied include S_2O_3'', S'', SO_3'', HSO_3', S_2O_5'' and S_2O_4'', which react at pH 4 to 10 and S_2O_6'', SO_4'' and S_2O_8'', which do not react at all. S_4O_6'' reacts only at pH $\simeq$ 10. S_2O_3'' is removed by Pb or Ba, the pH is adjusted to 4 to 5, any ppt. formed with $SbCl_3$ is removed, washed with cold H_2O and then NaOH is added to pH 10 at 80° C. Coloration can be observed with 8 μg per drop of test solution. Quantitative determination is by $KMnO_4$, the reaction being $5\ S_4O_6'' + 14\ MnO_4' + 21\ SO_4'' \rightarrow 27\ SO_4'' + 14\ MnSO_4 + 6\ O''$.

The most complex of possible mixtures is that with S_2O_3''. The S_2O_3'' is oxidised by alcoholic I to S_4O_6'', the S_4O_6'' being then determined by $KMnO_4$. The S_2O_3'' is separately determined and a correction is applied. Results are quoted for determinations in the presence of S'', SO_3'', S_2O_4'', S_2O_5'', S_2O_6'' and NO_2''.

W. C. WAKE

1507. **Determination by perchloric acid of chromium in one-bath chrome tanning liquors and in residual liquors of this tannage.** R. Durande-Ayme (*Bull. Ass. Franç. Chim. Ind. Cuir*, 1954, **16**, 11–13).—Cr can be accurately determined in chrome leather and in normal Cr liquors by Na_2O_2 and by other methods. Masking agents introduced into the liquors interfere with the results, but with these liquors, and with residual liquors containing organic matter, accurate determination is possible with $HClO_4$. Ten ml of test soln. are diluted with $\simeq$100 ml of distilled water. Fifteen ml of H_2SO_4 are added and then 4 ml of HNO_3 and 10 ml of $HClO_4$. The mixture is heated until it fumes, cooled and diluted, Cl is boiled off, KI is added, and the solution is titrated against 0·1 *N* $Na_2S_2O_3$, with starch as indicator. One ml of 0·1 *N* $Na_2S_2O_3 \equiv$ 0·002533 g of Cr_2O_3. B. R. HAZEL

1508. **Determination of tungstate.** W. C. Looney (*Dissert. Abstr.*, 1953, **13** [6], 974–975).—A gravimetric method is described for the determination of tungstate as tetraphenylarsonium metatungstate by the reaction of a soln. of $(C_6H_5)_4AsCl$ with a metatungstate soln. The pH is maintained between 1·5 and 4·0 with the aid of methyl orange, and a cryst. ppt. is obtained if the pptn. is carried out hot and is followed by digestion for 30–60 min. The ppt. is dried at 105° to 110° C and is weighed as $H_4[(C_6H_5)_4As]_4W_{12}O_{40}$ (factor for W 0·5034). Foreign ions that react with the reagent, precipitate metatungstate, reduce metatungstate, produce stable complexes or heteropoly tungstates under the conditions of the determination, or produce insol. hydrolysis products must be removed. Interference by Fe''', Cr''' and Al''' can be prevented by the introduction of a slight excess of PO_4''' ion. Several org. N compounds have been investigated as reagents for the quant. pptn. of tungstate. Quinoline, *iso*quinoline and 8-nitroquinoline have possible applications, but all three are inferior to cinchonine. A. JOBLING

1509. **Polarographic determination of tungsten in rocks.** L. E. Reichen (*Science*, 1954, **119**, 355–356).—A new polarographic wave for W in a supporting electrolyte of 4·6 *M* HCl and 0·1 *M* tartrate gives current - voltage curves not influenced by the constituents of the sample. The $E_{\frac{1}{2}}$ of the first wave is 0·35 V and that of the second 0·68 V. The sample is fused with Na_2CO_3, leached with water and filtered. The W is complexed with tartrate and the soln. is acidified with HCl. Vanadium, whose wave overlaps, is complexed with cinnamic acid and Fe is removed by filtration. Mo, Sn and Sb must be removed if their concn. is much greater than that of W. N. E.

1510. **Determination of uranium in concentrates by the fluoriphotometric method.** J. B. Zimmermann, F. T. Rabbitts and E. D. Kornelsen (*Canad. Dept. Mines and Tech. Surveys, Mines Branch, Techn. Paper No.* 6, 1953, 9 pp.).—The uranyl fluoride method of Zimmermann (*Chem. Abstr.*, **46**, 377a) can be used to determine U at concn. up to 90 per cent. of U_3O_8. Four sample portions are taken, and the final soln. of each is divided into 10 aliquots. A modified micro-pipette of the overflow transfer type is used to minimise errors. The 10 aliquots, an equal number of standards and duplicate blanks are fused at one time and measured immediately. The procedure is repeated for the remaining 3 samples, making a total of 88 determinations. Despite the large number of measurements that are made to give a good statistical average, the procedure is shorter than other recommended methods. Inter-laboratory comparisons show an average deviation between methods of less than 1 per cent. of the amount present. CHEM. ABSTR.

1511. **New argentimetric methods for the halogens.** J. Hernández Cañavate (*Inf. Quim. Anal.*, 1954, **8** [1], 1–10).—To obtain adsorption indicators that could be used in the highly acid media occurring in the argentimetric estimation of the halogens, a series of indicators of the type Fe^{III} - benzidine, Fe^{III} - *o*-tolidine and Fe^{III} - *o*-dianisidine was used. These redox systems behave in a reversible manner when the pH is sufficiently low. For the argentimetric estimation of chloride, NaCl solutions 0·1 *N*, 0·01 *N* and 0·002 *N* were prepared and 0·1 *N* and 0·01 *N* $AgNO_3$ solutions. *Procedure.*—To the chloride soln. add 8 to 10 drops of 4 per cent. iron alum soln., 1 ml of *N* nitric acid soln., and 5 drops of 1 per cent. soln. of *o*-tolidine in 95 per cent. ethanol containing 1 per cent. of glacial acetic acid. Silver nitrate soln. is added drop by drop with vigorous agitation in good illumination. The end-point is characterised by the decolorisation of the grey-blue or violet ppt. and the change from pale yellow to an intense canary yellow of the supernatant liquid.

The Fe^{III} - benzidine system behaves very similarly, but with Fe^{III} - *o*-dianisidine the supernatant liquid changes from pale pink to bright red at the end-point. Bromide and iodide were also estimated in the same way and the percentage error was determined; the greatest error occurred in weak solutions of the halogen (about 0·0001 *N*), when, with the iodide, there was an error of −0·66 per cent. In stronger solutions the error was small. H. PRITCHARD

1512. **Simplified potentiometric method for estimation of chlorides.** P. Deschamps (*Compt. Rend.*, 1954, **238** [1], 100–102).—Potentiometric titrations of water - acetone soln. of chlorides are made with 10^{-3} to 10^{-1} *N* $AgNO_3$ soln. with the aid of wire electrodes (1 mm diam.), one of Ag and the other Hg-coated Au. In chloride soln. $\simeq 10^{-2}M$ (for which the dead-stop end-point is used), the acetone concn. should be $\simeq 25$ per cent., increasing to 90 to 98 per cent. in 10^{-5} to 10^{-4} *M* Cl′ soln., then a few drops of 25 per cent. H_2SO_4 should be added and the circuit should be closed directly on galvanometer and 200-ohm resistance. All solutions are stirred mechanically. Procedures and method of obtaining and interpreting titration curves (galvanometer reading plotted against fraction pptd. by $AgNO_3$) are described for measurements in 10^{-5} to 10^{-2} *M* soln. of chlorides, alone or in presence of bromides. W. J. BAKER

1513. **Persulphate - cobalt method of determining manganese and chromium in steels and cast irons in one sample.** L. M. Kulberg, L. A. Molot and L. F. Grigoreva (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 370–372).—The accuracy and utility of the method of Kuznetsov and Budanova (*Brit. Abstr. C*, 1953, 383) are confirmed. After the titration of Mn by means of Na arsenite, Cr can be determined by titration with Fe^{II} salt in presence of N-phenylanthranilic acid. G. S. SMITH

1514. **The potentiometric determination of ferrous iron with potassium ferricyanide.** G. Wittmann (*Z. anal. Chem.*, 1954, **141** [4], 241–246).—Ferrous iron can be determined, even in the presence of arsenious acid, by a potentiometric technique and use of platinum - saturated calomel electrodes. Ferric ions must be added to prevent adsorption of ferrous ions on the ppt.; only then is the end-point independent of the HCl concn. To 20 ml of the unknown, add 100 ml of 12 per cent. HCl, 5 ml of $FeCl_3$ soln. (1 g of Fe per 100 ml) and titrate in the absence of oxygen with 0·1 *N* $K_3Fe(CN)_6$ soln. Zn, Cu, Pb, Mn^{II}, Co^{II}, Ni, Cd and Sb^{III} do not interfere, so the method is specially suitable for analysis of pyrites. P. S. STROSS

1515. **A new absorptiometric method of determining iron.** W. Nielsch and G. Böltz (*Z. anal.Chem.*, 1954, **141** [4], 247–252).—A new method of determining iron by the absorption at about 390 mμ of the iron - tartrate complex is described. The relation between colour formation and pH as well as tartrate concn. is investigated. At the optimum pH, 1·55 to 2·00, $NaIO_4$, is added as a stabiliser to prevent the photochemical reduction of the complex. If this pH is unsuitable for other reasons, measurements can be made at pH 5·00 to 5·75; no stabiliser is then necessary, but it does not interfere if present. To a soln. containing from 0·5 to 2·5 mg of Fe^{III} add 5 g of tartaric acid, 2 g of NH_4NO_3 and 10 mg of $NaIO_4$, make up to 100 ml and determine the extinction at about 390 mμ. Read the iron content from a calibration graph similarly constructed. P. S. STROSS

1516. **The application of the complexones in metallurgical analysis. [I. Ferrous materials.]** E. G. Brown (*Metallurgia*, 1954, **49**, 101–105).—The application of the complexones (ethylenediaminetetra-acetic acid and allied compounds) in metallurgical analysis is reviewed. In general their uses may be divided into four categories: masking action, colorimetric analysis, titrimetry and polarography. Examples of these functions are given and results of determinations of Co, Cr, Cu, Mn, Mo and Ni are tabulated. G. C. JONES

1517. **Spectrographic analysis of low-alloy steels.** British Iron and Steel Research Association (*Iron Steel Inst. Special Rep.*, No. 47, September, 1952, 83 pp.).—This report includes sections dealing with various general aspects of spectrographic analysis and concludes with a recommended method for low-alloy steels. The method proposed involves the excitation of the spectrum by means of a condensed spark discharge (with specified circuit parameters) between a pure graphite point and a flat surface prepared on the sample. A large-dispersion quartz spectrograph is used with normal photographic and photometric procedures. Calibration is effected by means of a series of carefully analysed standard samples, full details of which are given. The method is applicable to the analysis of low-alloy steels with iron contents of 95 ± 1 per cent., although corrections can be made to allow for compositions outside this range. The elements that can be determined and the concentration ranges covered are: silicon, 0·05 to 0·80; manganese, 0·05 to 1·50; nickel, 0·10 to 5·00; chromium, 0·05 to 3·00; molybdenum, 0·05 to 1·50; vanadium, 0·03 to 0·65; and copper, 0·05 to 0·50 per cent. The reproducibility of results obtained in several laboratories is given in detail for different concentration levels of the various impurities.
B. S. COOPER

1518. **Spectrochemical micro-analysis of steel and iron.** F. A. Pohl (*Mikrochim. Acta*, 1954, [2], 258–262).—For spectrochemical micro-analysis 1 to 10-mg samples of steel turnings are dissolved in 6·5 *N* HCl and the soln. is extracted with ether. As, Au, Fe^{III}, Ge, Ga, Mo, Sb, Sn and Tl^{III} are thus extracted. A small crystal of hydroxylamine and three drops of conc. HCl are added, and the ether is evaporated. To the residue is added 1 ml of *N* HCl and 0·1 ml of 20 per cent. NH_4CNS soln. Small amounts of solid sodium dithionite are added until the red colour has disappeared. The liquid is then extracted with ether. It is not possible to separate Au, As, Sb and Tl from the Fe by this method. The thiocyanate extracts are united with the first HCl phase and the elements contained in it are detected spectro-analytically at a high-voltage arc by the Feussner technique. The method can be used to detect Mn, Cu, Ni, Cr, V, W, Al, Ti, Zn, Sn and Zr, and, with some modification, could be used quantitatively.
A. J. MEE

1519. **Selection of line pairs for the spectrographic analysis of low-alloy steel.** D. L. Fry and T. P. Schreiber (*J. Opt. Soc. Amer.*, 1954, **44** [2], 159–162).—Suitable line pairs for the analysis of Mn, Cr, Ni, Mo, Si, Cu and V in low-alloy steel have been chosen on the basis of similarity of excitation potential and wavelength separation. Providing the excitation potentials of individual members of a pair are within ratios of 2 to 3 or 3 to 2 of each other, further equalisation of these potentials is of less importance in improving reproducibility than is reduction of wavelength separation.
B. S. COOPER

1520. **Influence of complex-forming ions on reducing potential of mercury. Analytical application to determination of ferric and ferricyanide ions.** F. Burriel [-Martí], F. Lucena-Conde and S. Bolle (*An. Soc. Esp. Fis. Quím., B.*, 1953, **49** [11], 693–700).—The reducing power of Hg is greatly enhanced by the presence of CNS′, and is then similar to that of Hg - HCl. Its most important reaction is reduction of $Fe^{\cdots}$, which can be applied to the determination of Fe. To reduce a 0·01 *M* soln. of $Fe^{\cdots}$ without pptg. $Hg_2(CNS)_2$, the CNS′ must be < 0·05 *M*. In alkaline soln. in presence of CN′, Hg acts as a powerful reducing agent. Atmospheric O is reduced to H_2O_2 and H_2O, and $Fe(CN)_6^{\cdots}$ is reduced to $Fe(CN)_6^{\cdots\cdot}$; the latter reaction can be used for quant. determination of Fe if air is excluded.
D. P. YOUNG

1521. **Mercurous salts as reductimetric reagents for titrations in alkaline medium. I. Titration of ferricyanide.** F. Burriel-Martí, F. Lucena-Conde and S. Arribas-Jimeno (*Anal. Chim. Acta*, 1954, **10** [4], 301–309).—Ferricyanide can be titrated according to the following reaction: $Fe(CN)_6^{\cdots} + Hg^{\cdot} + 4I' \rightarrow Fe(CN)_6^{\cdots\cdot} + HgI_4''$. The pH of the soln. must be < 14, the concn. of KI < 0·2 *M*, the temp. > 30° C and the pH of mercurous perchlorate soln. < 3·0. To a cold soln. containing the equiv. of 5 to 30 ml of 0·1 *N* ferricyanide add 25 ml of 4 *M* NaOH, 25 ml of water and 40 ml of *M* KI, and titrate immediately with 0·1 *N* $HgClO_4$, using Ba diphenylaminesulphonate as indicator or potentiometrically using a platinum wire and S.C.E. The max. errors noted are −0·2 per cent. and +0·4 per cent. The anions NO_3', Cl′ and SO_4'' do not interfere at concn. of 5 per cent.
W. C. JOHNSON

1522. **Determination of cobalt in metallurgical products.** J. Kinnunen, B. Merikanto and B. Wennerstrand (*Chemist Analyst*, 1954, **43** [1], 21–22).—Cobalt is determined colorimetrically as the thiocyanate, ascorbic acid being used to mask Fe, and thiourea to mask Cu. Dissolve 2·5 g of ore in HCl - HNO_3, add HF, and when decomp. is complete add 30 ml of dil. H_2SO_4 (1 + 1). Heat to fuming, cool and dilute. Filter into a 250-ml calibrated flask. Ignite the insol. matter, and fuse with $K_2S_2O_7$. Dissolve the melt in water and filter into the 250-ml flask. Dilute to the mark and mix. Pipette two 10-ml aliquots into two 100-ml calibrated flasks. Add a few drops of conc. NH_3 to produce a slight ppt., and redissolve in glacial acetic acid. Add 150 mg of ascorbic acid and 1 to 2 ml of sat. aq. thiourea soln. (or sufficient to make the soln. colourless). Add 20 ml of 50 per cent. aq. NH_4CNS to both, and 50 ml of acetone to one only. Make up to vol. and measure the optical densities at 625 mμ. An alternative digestion procedure is given, and for materials with large amounts of Fe and Cu, an extraction method with methyl *iso*-propyl ketone is proposed. Two determinations by the procedure described will not differ by more than ±0·01 per cent for purple ores containing about 0·60 per cent of Co.
G. B. THACKRAY

1523. **Spectrophotometric determination of cobalt in ores.** J. Gagnon (*Chemist Analyst*, 1954, **43** [1], 15–17).—The pink colour of $CoSO_4$ is used for the determination of Co in ores. The transmission curves of $CoSO_4$, $NiSO_4$, $CuSO_4$, $Cr_2(SO_4)_3$ are reproduced. Decompose a sufficient quantity of the sample to yield 8 to 150 mg of Co with HNO_3 - HCl-H_2SO_4 mixture. Heat to fuming, cool, dilute with 50 ml of H_2O and dissolve the salts. Add 10 ml of 50 per cent. aq. $Na_2S_2O_3.5H_2O$ and 10 ml of 10 per cent. aq. $Na_2PO_4.12H_2O$, and boil for 15 min. Cool, dilute to 100 ml, filter and measure the absorption at 520 mμ. Interfering elements include Cr, V, Mn (>50 mg), W (>30 mg), Fe (>400 mg), Ni(>30 mg); all amounts per 100 ml of final solution.
G. B. THACKRAY

1524. Colorimetric determination of cobalt by nitroso-R salt. III. Proposed method and its application. F. Burriel [-Martí] and R. Gallego (*An. Soc. Esp. Fís. Quím., B*, 1953, **49** [9–10], 587–590).—Two previous papers (*Brit. Abstr. C*, 1953, 158) have described the development of the method, which is suitable for the estimation of up to 45 μg of Co. Samples are treated with acid to destroy organic matter and then extracted with dithizone. The dithizone is destroyed and the complex of nitroso-R salt and Co is formed. The colour is compared with that of solutions of known Co content on two different types of absorptiometer. Results for Co in forage and in soil are given. H. H. M. JONES

1525. Use of diphenylthiocarbazone in analysis. IV. Instability constants of the dithizonates of nickel, cobalt, stannous tin, bismuth, cupric copper, silver and mercuric mercury. Dithizonates of manganese and iron. A. T. Pilipenko (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [5], 286–292).—Instability constants K' where, *e.g.*, for MDz_2—

$$K' = \frac{[M^{\cdot\cdot}]_{H_2O} \times [HDz]^2_{CCl_4} \times [K'_{HDz}]^2}{[MDz_2]_{CCl_4} \times [H^{\cdot}]^2_{H_2O}}$$

and $K'_{HDz} = 2 \times 10^{-9}$ (Babko and Pilipenko, *J. Anal. Chem., U.S.S.R.*, 1946, **1**, 275) have been determined for certain dithizonates with the following results: $NiDz_2$ $1{\cdot}7 \times 10^{-17}$, $BiDz_3$ $1{\cdot}1 \times 10^{-37}$, $HgDz_2$ $0{\cdot}7 \times 10^{-44}$, $SnDz_2$ $4{\cdot}5 \times 10^{-16}$, $CoDz_2$ 5×10^{-18}, $CuDz_2$ $1{\cdot}1 \times 10^{-27}$ and AgDz $2{\cdot}3 \times 10^{-18}$. $Fe^{\cdot\cdot}$ and Mn form dithizonates only under conditions at which oxidation rapidly occurs (pH > 7). The composition corresponds to 1 of metal to 2 of dithizone. G. S. SMITH

1526. Determination of nickel in plating solutions using disodium ethylenediaminetetra-acetate. K. E. Langford (*Electroplating*, 1954, **7** [2], 46–48).—A rapid and accurate volumetric method applicable to both dull and organic-type bright plating electrolytes is proposed for the determination of Ni in plating soln. Dilute 2 ml of plating soln. to 100 ml with water, add 10 ml of conc. NH_3 soln., 1 g of ammonium persulphate and 0·5 g of murexide and titrate the soln. with 0·2 *N* disodium ethylenediaminetetra-acetate to a bluish violet. If Co is present, boil the soln. for 3 min. after adding the ammonium persulphate and continue as before. The method is not recommended in presence of Mg. G. C. JONES

1527. Separation of platinum metals by ion exchange. I. The separation of palladium from iridium. W. M. MacNevin and W. B. Crummett (*Anal. Chim. Acta*, 1954, **10** [4], 323–327).—Palladium and iridium are separated by passage of an ammoniacal soln. of their chlorides through a column of Amberlite IR-100 cation-exchange resin. Palladium is retained as $[Pd(NH_3)_4]^{\cdot\cdot}$ whilst Ir passes through as $IrCl_6^{\prime\prime}$ or $IrCl_6^{\prime\prime\prime}$ and is eluted with a 0·025 *M* NH_3 - 0·025 *M* NH_4Cl soln. The Pd is recovered by elution with *M* HCl. W. C. JOHNSON

1528. Methods of spectrochemical analysis of trace metals. III. The analysis of trace metals in rocks and soils. F. A. Pohl (*Z. anal. Chem.*, 1954, **141** [2], 81–86).—A procedure is described for the concn. of trace metals in rocks and soils by extraction and for their spectrographic determination. The trace metals are separated from the alkali and alkaline-earth metals, SiO_2, Fe, Al and Ti. Silica is removed from the sample by treatment with HF and H_2SO_4 in a platinum crucible. The residue is dissolved in 6·5 *N* HCl and H_2O_2, and the Fe is separated by a combined chloride and thiocyanate extraction. The trace metals are then selectively extracted from the remaining constituents by means of $CHCl_3$ and ammonium tetramethylenedithiocarbamate at pH 3·5 to 4 and a $CHCl_3$ soln. of dithizone at pH 8 to 9. Cr (if present) is extracted more efficiently if it is first oxidised to the sexavalent state. After evaporating off the $CHCl_3$, dissolving the residue and adding Be, the solution is subjected to spectrographic analysis as described previously (*Brit. Abstr. C*, 1953, 516). By this procedure, Ag, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pd and V can be detected in a 1-g sample, the limit of detection being 1 μg. As little as 5 to 10 μg of Bi, Pb, Sn or Zn can be detected. As, Au and Tl accompany Fe in the thiocyanate; hence, if the Fe concn. is low, Fe need not be extracted and As, Au and Tl can be detected spectrographically. Samples rich in Mn interfere, but interference-free lines are quoted for use with small spectrographs. C. J. KEATTCH

1529. X-ray analysis of clays by Geiger - Müller counter. D. Schroeder (*Z. PflErnähr. Düng.*, 1953, **62**, 148–158).—A review, with 29 references. The advantages of the X-ray method over the visual and photometric methods are discussed. J. SCI. FOOD AGRIC. ABSTR.

1530. Quantitative determination of mullite in refractories by the X-ray method. Z. Ziołowski (*Prace Inst. Minist. Hutnic.*, 1954, **5** [6], 314–321).—The method described here is based on the photometric measurement of the intensity of X-ray lines recorded on film. A camera of the Bragg - Brennaro type is used, and the powder samples are placed in a rotating holder. Mullite, 96 per cent. pure, obtained from cyanite fired at 1500° C and purified by HF is used as a standard and NaCl (a 10 per cent. addition) as a reference substance. The analytical curve for determination of mullite shows the percentage of mullite as a function of the intensity ratio of the mullite (121) line ($d = 0{\cdot}22$ mμ) and the NaCl (220) line ($d = 0{\cdot}199$ mμ). The max. error of the measurements in the 20 to 80 per cent. mullite range is $\simeq 14$ per cent. S. K. LACHOWICZ

1531. Functional group analysis: characterisation of coal hydrogenation products. R. A. Glenn and E. D. Olleman (*Anal. Chem.*, 1954, **26** [2], 350–352).—Functional group analysis is combined with ultimate analysis and mol. wt. determination for the determination of various types of O and N compounds in coal hydrogenation neutral oils. Acid and alkaline (aq. and alcoholic) extraction of the neutral oil and analysis of the extracts is followed, on the residual oil, by sodium aminoethoxide titration, $HClO_4$ titration, analytical acetylation, analytical acetylation followed by selective hydrolysis, and treatment with a Grignard reagent. From these results the proportions of most of the functional groups are deduced; ethers, N-substituted pyrroles and hydrocarbons are found by difference. D. BAILEY

See also Abstracts 1449, 1450, 1452, 1454, 1455, 1456, 1457, 1532, 1539.

3.—ORGANIC ANALYSIS

1532. **Determination of deuterium in organic compounds.** M. Corval and R. Viallard (*Mikrochim. Acta*, 1954, [2], 231–239).—The substance is burned in a current of dry air or O, combustion being brought about by passing the mixture over an oxidation catalyst at 400° to 420° C, as in the ter Meulen technique. For compounds containing C, H and O, a specially prepared form of MnO_2 is used. The H_2O formed is condensed in a vessel immersed in CO_2-snow, and it is later distilled *in vacuo* into a known volume of H_2O of known density. The density of the mixture is then determined by flotation, and the amount of D_2O is calculated. A. J. MEE

1533. **Volumetric estimation of [nitrogen in] organic bases by titration with tellurium tetrabromide.** P. Dupuy and M. Nortz (*Compt. Rend.*, 1954, **238** [5], 587–588).—The method involves titration of $\simeq$10 ml of 0·1 *N* $TeBr_4$ in an acetic acid - ethanol mixture with a soln. of an org. amine in acetic acid, the exact disappearance of the deep-orange colour of $TeBr_4$ (which forms colourless complexes with org. bases) being indicated by means of a few drops of a conc. soln. of $CuCl_2$ or $CoBr_2$ in ethanol. The prep. of the $TeBr_4$ soln. and its standardisation against an acetic acid soln. of aniline are described. The method is applicable in presence of aliphatic acids, alcohols, etc., and is claimed to be accurate to $\pm$1 per cent of N. W. J. BAKER

1534. **A new method of simultaneous microdetermination of fluorine, hydrogen and carbon in organic compounds.** N. E. Ghel'man and M. O. Korshun (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1953, **89** [4], 685–687).—The material in a normal C and H micro-analysis is covered with MgO, which reacts with fluorine released from the compound (to yield MgF_2), but does not absorb CO_2 and H_2O. R. C. MURRAY

1535. **Some observations on the C-methyl determination.** E. J. Eisenbraun, S. M. McElvain and B. F. Aycock (*J. Amer. Chem. Soc.*, 1954, **76** [2], 607–609).—A quant. study has been made of the Kuhn - Roth C-methyl determination. Values are tabulated of the found C-methyl value, the amount of oxidising agent (Cr_2O_7'') actually consumed in the reaction, and the amount of Cr_2O_7'' theoretically required for oxidation to specified degradation products for each of 69 org. compounds. There are wide variations in the ratio of found to theoretical consumption of Cr_2O_7''. A. JOBLING

1536. **A colour-reaction for detection of methylenedioxy groups.** O. R. Hansen (*Acta Chem. Scand.*, 1953, **7** [7], 1125).—On heating substances containing methylenedioxy groups with chromotropic (1:8-dihydroxynaphthalene-3:6-disulphonic) acid in 90 per cent. H_2SO_4 a purple coloration is formed owing to reaction between the reagent and liberated formaldehyde. The coloration is not destroyed on dilution of the H_2SO_4, unlike the colours formed by H_2SO_4 with many organic substances. C. E. SEARLE

1537. **Analysis for industry. Determination of acetyl groups.** J. H. Thompson (*Ind. Chem.*, 1954, **30**, 127–129).—Straight hydrolysis methods and transesterification procedures (particularly semi-micro and micro methods) are reviewed, including saponification with toluene-*p*-sulphonic acid and with tungstophosphoric acid. J. M. JACOBS

1538. **Rational process for verifying the composition of compounds from analytical data as proposed by P. Eynbrodt.** N. P. Komar (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 373-376). The method of Eynbrodt (1848), slightly modified, is recommended for checking the formula, derived from analytical data, of an organic compound. Calculate a coeff. q for each element equal to (mol. wt. of compound × percentage of the element found) ÷ (number of atoms of this element in the formula × at. wt. of the element × 100). With correct analysis and correct formula, all values of q must be unity. Examination of the departures from unity will show if the formula is probably correct or, if probably incorrect, where correction may have to be made. G. S. SMITH

1539. **Polarographic determination of traces of metals in organic material. Determination of Pb, Cu, Cd, Ni, Zn and Fe.** E. Wåhlin (*Acta Chem. Scand.*, 1953, **7** [6], 956–968).—A method is described for the rapid routine analysis of Pb, Cu, Cd, Ni, Zn and Fe in organic materials with a standard deviation of $\pm$2 at 50 to 100 p.p.m. and 0·4 at 2 p.p.m. Two 5-g samples of cellulose, carboxymethylcellulose or dried yeast are subjected to a wet combustion with a mixture of conc. HNO_3 and conc. $HClO_4$. NaCl is sometimes added to give a residual salt cake, which facilitates solution of the trace metals remaining after evaporation of the acids. Pb in one sample is determined polarographically in acid soln. after pptn. by aq. NH_3 together with $Fe(OH)_3$. Fe in the second sample is determined polarographically in $HClO_4$ solution; $2NH_2OH.H_2SO_4$ and pyridine are then added, and Cu, Cd, Ni and Zn are determined. C. E. SEARLE

1540. **Application of ion-exchange resins to organic analysis.** B. S. Miller and J. A. Johnson (*Trans. Amer. Ass. Cereal Chem.*, 1954, **12** [1], 29–42).—A brief resumé is given of the various types of organic ion-exchange materials and their functional differences in structure, together with their methods of use and principal applications in (*i*) removal of impurities, (*ii*) adjustment of pH and (*iii*) separation and subsequent analysis of mixtures of related compounds. Separations of amino-acids and of mixtures of organic acids, alcohols, aldehydes and ketones are described. M. TADMAN

1541. **Paper-chromatographic separation of alcohol - water mixtures.** J. Sivadjian (*Compt. Rend.*, 1954, **238** [6], 678–680).—It is claimed that a satisfactory separation of a binary mixture of aliphatic alcohol and H_2O can be effected by placing a few drops of the mixture on filter-paper, which is then pressed firmly between two photographic plates, whereby H_2O enters the gelatin of the latter forming a grey spot ($\simeq$16 mm diameter) with a black border, while the alcohol forms a normal but sometimes larger spot ($\simeq$20 to 30 mm diameter) on the paper. Results obtained with *n*- and *iso*-propanols and *tert.*-butanol containing various amounts of H_2O indicate the degree of separation achieved. W. J. BAKER

1542. **The preparation of S-alkylthiuronium picrates and a new method for the estimation of tertiary alcohols.** L. Schotte and S. Veibel (*Acta Chem. Scand.*, 1953, **7** [10], 1357–1363).—A number of tertiary alcohols are converted to their chlorides

with conc. HCl. Refluxing with aq. alcoholic thiourea yields the corresponding S-alkylthiuronium chlorides, which are then converted to their picrates (melting-points are recorded). Alcohols containing phenyl groups do not react under the prescribed conditions. The equivalents of the tertiary alcohols can be determined with an error of <2 units by titration of the picrates electrometrically or with crystal violet as indicator.

C. E. SEARLE

1543. **Recognition of polyalcohols with the vanadium - hydroxyquinoline complex and general application of the new reagent to recognition of alcohols in macro- and semimicro-analysis.** F. Buscaróns, J. L. Marín and J. J. Claver (*An. Soc. Esp. Fís. Quím., B*, 1953, **49** [5], 367–374).—The vanadium - oxine reagent (*Brit. Abstr. C*, 1949, 386) is applicable to polyols only if they are sol. in inert org. solvents or can be concentrated to 50 per cent. or more in water.

D. P. YOUNG

1544. **Polarographic determination of aldehydes and ketones in base solutions of semicarbazide.** P. Souchay and M. Graizon (*Chim. Anal.*, 1954, **36** [4], 85–91).—The polarographic waves given in base solutions of semicarbazide hydrochloride (**I**) by certain aldehydes and ketones, particularly those difficultly electro-reducible in other media, have been found suitable for the determination of these substances alone and, in certain circumstances, in mixtures. *General procedure*—To 25 ml of *M* **I** add *x* ml of *M* NaOH and dilute to 50 ml. Remove air from 25 ml with hydrogen and add 1 ml of 0·026 *M* ethanolic soln. of ketone or aldehyde and run the polarogram from −0·7 V. For ketones, max. height of step is at pH 4·0 ($x = 4·0$) and for aldehydes at pH 1·8 ($x = 0$). Add Tylose where necessary to suppress max. Differences in $E_{\frac{1}{2}}$, which varies with pH, may allow determinations of mixtures in some instances, *e.g.*, acetone - acetophenone or acetone - benzaldehyde in a base soln of 0·5 *M* **I** and 0·3 *M* NaOH. Aldehydes may usually be determined in the presence of ketones in acid soln. with low concn. of **I**, *e.g.*, 0·1 *M* HCl and 0·02 *M* **I**. For determining ketones in the presence of aldehydes, the mixture is treated with Ag_2O (Mitchell *et al.*, *Brit. Abstr. C*, 1950, 402) to remove aldehydes and aliquots are added to base soln. of 0·5 *M* **I** and 0·3 *M* NaOH.

H. F. W. KIRKPATRICK

1545. **The separation of 2:4-dinitrophenylhydrazones.** G. A. Howard and A. R. Tatchell (*Chem. & Ind.*, 1954, [8], 219).—Separation of the 2:4-dinitrophenylhydrazones of certain mixtures of several simple carbonyl compounds is achieved by means of reverse-phase partition column chromatography, with benzene as the mobile and formamide as the stationary phase in a column packed with kieselguhr that has been pre-treated with dimethyldichlorosilane.

D. A. PANTONY

1546. **Inverse retention. A method for the quantitative estimation of paper-chromatographically separated organic acids.** E. R. Reichl and J. E. Löffler (*Mikrochim. Acta*, 1954, [2], 226–230).—In the method of inverse retention, the acid solution is allowed to penetrate into the reagent fixed on the paper. An aq. soln. of basic Pb acetate is used as the pptg. agent. The chromatogram is developed with an acetone - H_2O mixture, the composition of which varies according to the acid to be determined. Before use, the acetone is neutralised to phenolphthalein with NaOH and distilled. The paper is prepared by washing with 10 per cent. acetic acid and drying. The test soln. is placed on the paper to form a number of strips. The paper is then eluted with an acid-solvent mixture. A mixture of ethyl acetate, acetic acid and H_2O in the proportions 14:3:3 is suitable. After drying, the paper is turned through 90° and folded about 1 cm from its middle. The crease is immersed to a depth of 1 to 2 mm in molten diphenyl. When the diphenyl has solidified, the upper (acid-free) half is immersed up to the diphenyl strip in freshly prepared basic Pb acetate soln., dried between layers of filter-paper, and heated to 110° C until there is no smell of diphenyl. The completely dry and diphenyl-free paper is rolled into a cylinder and placed in a dish with the acetone - H_2O mixture. After about 2 hr. when the mixture has reached the upper edge of the paper, it is dried. This process may be repeated a second time in order to ensure that the acid has risen completely. The acid spot is made visible in the retentiogram by $AgNO_3$ - phenol. The white retention spots are cut out, dried and weighed. The weight is multiplied by a factor found by using the procedure with solutions of known concn. The numerical value of this factor is the same for all the acids investigated; a spot weight of 100 mg corresponds to 0·47 μ mol. of acid. Aconitic, malic, citric and gallic acids can be determined simultaneously with the same solvent. The mean error is ± 6 to ± 12 per cent. depending on the acid. The lead salts of the acids must be insol. in the acetone - H_2O mixture. Weights of acid between 10 and 150 μg can be determined in solutions that may contain as little as 0·03 per cent. of the individual acids. The method can be used successfully with plant extracts.

A. J. MEE

1547. **Colorimetric micro-determination of citric and *cis*-aconitic acids.** J. Nekshorocheff and J. Wajzer (*Bull. Soc. Chim. Biol.*, 1953, **35** [7], 695–696).—The reaction (Fuerth and Herrmann, *Biochem. Z.*, 1935, **280**, 448) of several tricarboxylic acids with acetic anhydride and pyridine has been found to give a colour in the visible part of the spectrum which varies according to the conditions. By operating with the crystalline acids or on the dry residue from the evaporation of a soln., a constant absorption is observed with a max. between 360 and 380 mμ. Citric acid gives the reaction after heating with the reagent, *cis*-aconitic acid in the cold; hence the method can be applied to the determination of these acids separately.

N. E.

1548. **Quantatitive determination of keto-acids by paper partition chromatography.** D. Cavallini and N. Frontali (*Biochim. Biophys. Acta*, 1954, **13** [3], 439–445).—The method previously reported (*Brit. Abstr. C*, 1950, 90 and 402) has been simplified, special attention being paid to the sensitivity and accuracy of the quant. determination of pyruvic and α-oxoglutaric acids in biological material. The keto-acids are converted into the 2:4-dinitrophenylhydrazones, which are applied to the paper in aq. NH_3. The developing solvent is *n*-butanol - ethanol - water (40:10:50). The spots are extracted with *N* NaOH and their light absorptions are determined. The recovery of pyruvic and α-oxoglutaric acids averages 94 per cent.

N. E.

1549. **Reaction between chlorous acid and glucose. Quantitative stoicheiometry and evaluation of reagent decomposition.** H. F. Launer and Y. Tomimatsu (*Anal. Chem.*, 1954, **26** [2], 382–386).—The oxidation of glucose by chlorous acid is treated

theoretically and the concurrent decomposition of the reagent during the oxidation is evaluated. Sodium chlorite in phosphoric acid - phosphate buffers over the range pH 2·4 to 3·4 reacts with glucose in the ratio 3 to 1 without over-oxidation with time. Reagent decomposition, which is sensitive to light and, to a lesser extent, to ionic strength, during oxidation of glucose, is evaluated by assuming that its rate is proportional to the geometrical mean of chlorite concentration. A simple analytical expression is developed which holds over the ranges 0·000004 to 0·0003 *M* glucose, 0·0005 to 0·0032 *M* chlorite and pH 2·4 to 3·4 at 50° C. D. BAILEY

1550. **Separation of glucose and sorbitol by paper chromatography.** S. N. Parikh, J. M. Parikh and A. N. Godbole (*Curr. Sci.*, 1954, **23** [2], 53).—Glucose and sorbitol are separated by the following developing solvents: *m*-cresol - phenol - water, 4:2:1 by vol. (R_F sorbitol 0·24 ± 0·01, glucose 0·16 ± 0·01), the same liquids, 2:2:1 by vol. (R_F sorbitol 0·31 ± 0·01, glucose 0·22 ± 0·01) or phenol - water, 4:1 by vol. (R_F sorbitol 0·52 ± 0·02, glucose 0·41 ± 0·02). The spray reagent, a mixture of 0·1 *M* boric acid (33·3 ml), 0·1 *M* NaOH (26·7 ml) and 0·02 per cent. aq. alcoholic methyl red (40 ml), gives red spots on a yellow background. D. BAILEY

1551. **Specific method of determination of carbonyl groups in hydroxycelluloses.** E. D. Kaverzneva and A. S. Salova (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 365–369).—The normal hydroxylamine method for determining carbonyl groups in hydroxycellulose gives results that are too high because of the simultaneous reaction of lactone groups to give hydroxamic acids. The lactone reaction can be eliminated by working at pH 3 to 4 in alcoholic medium. G. S. SMITH

1552. **The determination of secondary amines as nickel - dithiocarbamate complexes.** L. Nebbia and F. Guerrieri (*Chim. e Ind.*, 1953, **35** [12], 896–899).—A mixture of primary, secondary and tertiary amines is treated with CS_2 in *iso*pentanol to convert the first two to dithiocarbamates, which are then converted to complexes with $NiSO_4$; the dithiocarbamates of primary amines are then removed by dissolving them in alkali. For small quantities of secondary amines the $NiSO_4$ complex is decomposed with $AgNO_3$, and for large quantities with HNO_3, the liberated Ni being titrated with ethylenediaminetetra-acetic acid in the presence of murexide. In this way 100 μg to 0·5 g of secondary amines can be determined. R. C. MURRAY

1553. **Determination of phenanthrene in coal-tar products.** L. Blom and W. J. Vranken (*Anal. Chem.*, 1954, **26** [2], 404–407).—A modified procedure is described for the determination of phenanthrene in coal-tar. A soln. of 2·5 g of the sample in 100 ml of glacial acetic acid is treated whilst hot with 3·4 ml of a mixture of equal parts of conc. HCl and 40 per cent. aq. formaldehyde, refluxed for 15 min., filtered and diluted with acetic acid to 200 ml. This soln. (20 ml) is added slowly to a boiling mixture of 6 g of iodine pentoxide, 10 ml of acetic acid and 70 ml of water and boiling is continued for 10 min. The cooled soln. is treated with 45 ml of saturated $NaHSO_3$, warmed to 55° C, diluted with 125 ml of water, cooled, filtered and treated with 20 ml of a 4 per cent. soln. of *o*-phenylenediamine dihydrochloride. After refluxing for 25 min. followed by heating for 15 min. on a steam-bath, the mixture is treated with 3 g of trichloroacetic acid and the ppt. of phenanthrophenazine is filtered, washed and dried. Carbazole interferes and must be removed before oxidation; acenaphthene, indole and phenolic substances are removed by the pre-treatment with formaldehyde, whilst naphthalene, anthracene, fluorene and pyridine bases do not interfere. D. BAILEY

1554. **The determination of o-cresol, 4-chloro-2-methylphenol and 2-methylphenoxyacetic acid by quantitative bromination.** P. Aichenegg and H. G. Haynes (*J. Appl. Chem.*, 1954, **4** [3], 137–140).—The Koppeschaar "bromination-excess" method is applied to the rapid estimation of *o*-cresol, 4-chloro-2-methylphenol and 2-methylphenoxyacetic acid. A soln. of the sample in NaOH or ethanol is strongly acidified with HCl, a small excess of aq. 0·1 *N* $KBrO_3$ - KBr is added quickly and the reaction is allowed to proceed for ≃3 min., after which the excess of Br is estimated iodimetrically. An alternative "direct-bromination" method in which the end-point is revealed electrometrically is also described—a permanent galvanometer displacement occurs when the cathode of two platinum wires, immersed 1 mm apart in the acidified soln., becomes depolarised by free Br. Both methods are applicable in presence of 4:6-dichloro-2-methylphenol and chlorinated 2-methylphenoxyacetic acids, but slight modifications are necessary when chlorinated hydrocarbons or nitrobenzene are present. W. J. BAKER

1555. **Determination of phenols in waste waters by ultra-violet absorption.** L. J. Schmauch and H. M. Grubb (*Anal. Chem.*, 1954, **26** [2], 308–311).—The determination of phenols in refinery waste water by a spectrophotometric method based on the shift with pH of the u.v. spectra of phenols is described. The water sample is adjusted to pH 12 with solid KOH and extracted with CCl_4 to remove oil. The oil-free sample is then adjusted to pH 5 with HCl and extracted with tributyl phosphate to recover the phenols as a concentrate. This concentrate is divided into two portions, one of which is returned to a pH value of 12 by treatment with 0·1 *M* methanolic trimethyloctadecylammonium hydroxide and the other is diluted with a corresponding volume of methanol. Spectrophotometric comparison at 301 mμ of the two portions gives a measure of the phenols by the bathochromic-difference technique. An absorbance of 0·015 per cm for 0·01 p.p.m. of phenols in the sample is obtained. Contributions of naphthols and dihydric phenols are appreciable, whilst thiophenol has a much smaller effect. Basic N compounds, carboxylic acids and sulphonic acids, which have spectra insensitive to pH, show insignificant effects. The accuracy of the method is better than 80 per cent. and although more reliable than colorimetric procedures is more time-consuming. D. BAILEY

1556. **Detection of piperonal in mixtures of vanillin and piperonal.** A. Castiglioni and G. Bionda (*Z. anal. Chem.*, 1954, **141** [3], 189–190).—Piperonal can be detected in the presence of vanillin on the micro scale by the formation of an insol. condensation product with *cyclo*hexanone. A 0·1-ml portion of the test soln. is reacted with 0·2 ml of a 10 per cent. soln. of *cyclo*hexanone in 95 per cent. ethanol, and 2 ml of 10 per cent. aq. NaOH is added. After shaking, the soln. is brought to the boil and set aside. If piperonal is present, the liquid becomes turbid in 15 min. and turns

milky, but a ppt. may separate immediately. As a confirmation, the liquid is filtered, the ppt. is washed with water and the filter is dried. On the addition of a drop of H_2SO_4, the deep violet colour of 2:6-dipiperonylidene*cyclo*hexanone confirms the presence of piperonal. Vanillin forms no ppt. under these conditions with *cyclo*hexanone, but gives a yellow-red soln. which may make the turbidity less easy to observe. Coumarin, salicylic acid, acetylsalicylic acid and benzoic acid in the vanillin do not interfere. The sensitivity of the method is $\simeq 0{\cdot}0001$ g of piperonal.

J. H. WATON

1557. **A note on the methoxyl determination of methylated derivatives of ellagic and hexahydroxydiphenic acids.** W. Mayer (*Z. anal. Chem.*, 1954 **141** [5], 345–347).—In ordinary Zeisel determinations on methylated derivatives of ellagic acid and hexahydroxydiphenic acid results are low. When the determinations were carried out by the Pregl-Roth micro-method, it was found that the results varied with the amount of phenol used as solvent. With 5 to 10 times the normal amount of phenol correct results are obtained; the low results are due to the pptn. of partly demethylated derivatives, which are less soluble in phenol than the fully methylated compounds. E. HAYES

1558. **Detection of small quantities of acetone in furfural.** G. Bionda (*Chim. e Ind.*, 1954, **36** [2], 110).—Two to five ml of furfural are covered with the same vol. of aq. or aq.-alcoholic NaOH; the presence of $>0{\cdot}02$ per cent. of acetone in furfural is shown by a yellow turbidity in the NaOH, caused by formation of difurfurylideneacetone. With $<0{\cdot}02$ per cent. of acetone, an intense red-violet colour is given when the difurfurylideneacetone is filtered off and treated with 60 per cent. H_2SO_4.

R. C. MURRYA

1559. **Determination of furfural in oil in refinery operations by ultra-violet spectrophotometry.** L. L. Gent, R. C. Pomatti and H. Levin (*Anal Chem.*, 1954, **26** [2], 413–414).—Furfural is determined in oil by dilution with *iso*octane, extraction with aq. $NaHSO_3$ and spectrophotometric measurement after alkali decomp. of the $NaHSO_3$-furfural addition product. The oil sample, containing 0·00005 to 0·001 g of furfural, is diluted with 50 ml of *iso*-octane and shaken for 5 min. with 20 ml of 10 per cent. aq. $NaHSO_3$ soln. After separation of the layers, 10 ml of the aq. layer are removed, treated with 15 ml of 10 per cent. aq. KOH and diluted to 50 ml. The absorption of this soln. is measured at 277 mμ, the absorption of the blank is deducted and the furfural content is found by comparing the net absorption with those of standards.

D. BAILEY

1560. **Paper chromatographic separation of the isomeric pyridinecarboxylic acids.** T. Hashizume (*Nature*, 1954, **173**, 645).—Pyridine monocarboxylic acids can be separated by paper chromatography by a solvent mixture of *n*-butanol and methanol containing 35 per cent. v/v of water. After development, the solvent is evaporated at room temp. and the strips are sprayed with a 0·2 per cent. soln. of bromocresol purple in *n*-butanol-water, giving yellow spots on a blue background. Under these conditions the R_F values of the acids are quite reproducible, decreasing in the order $\beta > \gamma > \alpha$. In a mixture of the three, the R_F values are slightly lower than with the individual acids. H. P. PAGET

1561. **Colorimetric method for quantitative micro-determination of quaternary ammonium compounds. Losses of quaternary ammonium compounds caused by glass adsorption and concentration in the foam.** J. Fogh, P. O. H. Rasmussen and K. Skadhauge (*Anal. Chem.*, 1954, **26** [2], 392–395).—A spectrophotometric method of analysing cetylpyridinium chloride in concn. ranging from 0 to 25 μg per ml is described. It is based upon the ability of bromocresol purple to produce a blue colour with different quaternary ammonium compounds at pH 8·2. The method is independent of variations in temp. within certain limits and uninfluenced by the presence in the test solutions of Ca, Mg, Fe^{II}, Fe^{III} and Cu^{II} at concn. up to 1 per cent. The tendency for the quaternary ammonium compound to be adsorbed on glass (especially if badly scratched) is demonstrated, and losses, caused in this way, of cetylpyridinium chloride from a soln. of concn. 1 in 50,000 vary from 0 to 70 per cent. with variation in the glass surface. By treating glass surfaces with Plexiglas, adsorption can be diminished to a great extent. After shaking a soln. of cetylpyridinium chloride, an appreciable increase is observed in the concn. of the quaternary ammonium compound in the foam phase, the liquid phase containing 9·8 per cent. less than before shaking. D. BAILEY

1562. **Refractive indices of some morpholine solutions.** C. M. Wheeler, jun., and C. G. Houle (*Anal. Chem.*, 1954, **26** [2], 414–415).—Refractive indices are reported for pure components and binary solutions of morpholine with aniline, benzene, ethanol or water at various concn. D. BAILEY

1563. **Chromatographic fractionation of crude petroleum oils.** C. Karr, jun., W. D. Weatherford, jun., and R. G. Capell (*Anal. Chem.*, 1954, **26** [2], 252–256).—The application of the technique of elution chromatography to the fractionation of undiluted virgin crude oils is described. Data are presented demonstrating the type and extent of fractionation obtained on alumina and bauxite adsorbents and three eluting solutions (*n*-pentane, thiophen-free benzene and 25 per cent. v/v ethanol in thiophen-free benzene) of increasing elutive power. The alumina and bauxite adsorbents are satisfactory for separating hydrocarbon components from high-S crude oils in a colourless, S-free, metal-free, fractionated state, while yielding S-compound concentrates and asphaltic fractions. Certain hydrocarbon classes, such as paraffins and mononuclear aromatics, are recovered directly uncontaminated by other hydrocarbon classes by proper selection of fractions. An apparatus for fractionating virgin crude oils by semi-continuous elution is described and illustrated. D. BAILEY

1564. **Rapid determination of aromatics in petroleum fractions. Absorption with picric acid-nitrobenzene.** E. A. Pasquinelli (*Anal. Chem.*, 1954, **26** [2], 329–342).—A rapid and simple method is described for determining the aromatics in a wide variety of petroleum fractions boiling below 600° F. The petroleum sample (10 ml) is shaken for 2 min. at $20° \pm 0{\cdot}5°$ C with an amount of picric acid-nitrobenzene reagent (217·5 g of anhyd. picric acid in 1 litre of soln.) determined by the mean mol. wt. of sample. After shaking, the tube is stood vertically for 10 min. until the position of the meniscus is no longer changing; the absorption of the liquid is then determined. The method, which is specific for aromatic hydrocarbons, has a precision of $\pm 0{\cdot}5$ per

cent. and an accuracy of ±1 per cent. A qual. test is also described in which 20 ml of sample are shaken with 1 ml of reagent for 10 sec. After 10 min. the colour of the clear liquid is observed. D. BAILEY

1565. **Determination of hydrocarbon gases as air contaminants.** E. R. Quiram, S. J. Metro and J. B. Lewis (*Anal. Chem.*, 1954, **26** [2], 352–354).—A method is described for the determination of small quantities of gaseous hydrocarbons in the atmosphere. Dry, contaminated air (2 to 5 cu. ft.) is drawn over silica gel at −100° F (this temp. is produced by the use of solid CO_2 and *iso*propanol) to remove the gaseous hydrocarbons. These are then desorbed from the gel by heating, collected at low pressure and analysed on a mass spectrometer. Analysis of synthetic mixtures shows that C_3, C_4 and C_5 hydrocarbons are completely retained (95 per cent. recovery), but the recovery of C_2H_6 is only 60 per cent. D. BAILEY

1566. **Analysis of hydrocarbon oils based on density, refractive index and aniline point. II. Estimation of naphthenic carbon.** L. Robert (*Rev. Inst. Franç. Pétrole*, 1953, **8** [12], 586–589).—The formula giving the percentage of aromatic carbon (A) in an oil as a linear function of n_D, d and aniline point (P) applies only when $A < 30$ (*cf. Brit. Abstr. C*, 1952, 338). For oils containing more aromatics, a corrected value ($0{\cdot}5A + 15$) is taken. A similar formula gives the percentage of naphthenic carbon (N) with a precision within ±3 per cent., *viz.*,

$$N = 1573{\cdot}3\ n_D^{20} + 840{\cdot}15\ d_4^{20} - 0{\cdot}4619\ P + 1666{\cdot}2.$$

The proportion of paraffinic carbon is found by difference. The mean mol. wt. (M) is given approximately by

$$M = 1705{\cdot}45\ n_D^{20} + 792{\cdot}93\ d_4^{20} + 4{\cdot}553\ P - 3287.$$

A. R. PEARSON

1567. **A method for the estimation of Fibrolane BX or viscose rayon in blends with wool.** A. N. Davidson and R. Preston (*J. Text. Inst.*, 1954, **45** [2], T142–T146).—A method described for the estimation of Fibrolane BX or viscose rayon blended with wool is based on the preferential solubility of wool in a boiling soln. prepared by dissolving 5 g of hydrated $Ba(OH)_2$ and 20 g of KNO_3 in 100 ml of water. After boiling for 6 min., 80·6 per cent. of the Fibrolane BX and 100 per cent. of the viscose rayon are recovered. L. VALENTINE

1568. **The determination of lignin in partially delignified jute.** W. G. MacMillan, A. B. Sen Gupta and A. Roy (*J. Text. Inst.*, 1954, **45** [2], T 108–T 112).—The lignin contents of partially delignified jute, as determined with 72 per cent. H_2SO_4 for 48 hr. at 2° C or 1 hr. at 22° C, are almost the same as the calculated values obtained by applying the correction factors necessary to take account of the partial solubility of lignin in H_2SO_4 and the presence of impurities in the lignin (*J. Text. Inst.*, 1952, **43**, T103). As a result, the rapid method (1 hr. at 22° C) can safely be applied to the determination of the lignin content of partially delignified jute. L. VALENTINE

1569. **An absorption method for semi-quantitative determination of coloured materials in very dilute solution.** S. Končar-Djurdjević and S. Joksimović-Tjapkin (*Anal. Chim. Acta*, 1954, **10** [4], 346–355).—A method is described for the semi-quantitative determination of coloured substances in concn. too small to be measured directly either optically or by photo-electric instruments. The soln. is passed through a septum of filter-paper coated with a suitable adsorbent material, and an equilibrium is established between the soln. and the adsorbent. The intensity of colour on the adsorbent surface is measured photo-electrically by reflected light. Methylene blue in concn. 10^{-5} to 10^{-7} per cent. is adsorbed on filter-paper sprayed with a suspension of silica gel (100 mesh) in cellulose nitrate soln.; when the photo-electric current is plotted against log (concn.), a straight line is obtained. Congo red in similar concn. is adsorbed on filter-paper sprayed only with cellulose nitrate to diminish the rate of flow and to strengthen the paper; for this dye a straight line is obtained on a log-log graph. W. C. JOHNSON

1570. **The identification of lipstick dyes by paper chromatography.** J. Deshusses and P. Desbaumes (*Mitt. Lebensm. Hyg., Bern*, 1953, **44** [6], 500–507).—The dyes are extracted by hot 50 per cent. acetic acid and the cooled acid soln. is filtered. The filtrate is treated with light petroleum to remove fatty material and then evaporated to dryness. The residue is taken up in 50 per cent. ethanol and chromatographed on Whatman No. 1 filter-paper by an ascending technique; the solvent consists of 96 per cent. ethanol (80 ml), water (112 ml) and 25 per cent. NH_3 (8 ml). R_F values for several dyes are given, as well as results obtained on commercial lipsticks. E. HAYES

1571. **Analytical use of the halochrome effect. I. Interaction of antimony trichloride with some dyes.** L. M. Kul'berg, I. S. Mustafin and A. I. Cherkesov (*Ukr. Chem. J.*, 1952, **18** [6], 641–645).—The blue colour produced by reaction (Isakov) of $SbCl_3$ with Sudan-III can be obtained with other dyes including phthaleins; it is caused by a halochrome effect. A method of detecting traces of $SbCl_3$ in $SnCl_2$, based on such a reaction with thymolphthalein, is proposed. R. C. MURRAY

1572. **A qualitative analysis of synthetic fibres.** M. Lundegard and E. D. Roseberry (*Amer. Dyest. Rep.*, 1954, **43** [4], 93–97).—Simple, accurate and reproducible procedures for identification of Nylon, Dacron, Orlon, Acrilan, X-51, Dynel, Saran and polyethylene by tests involving (*i*) chlorine detection, (*ii*) flame test, (*iii*) chemical reactions including the Lieberman-Storch test, (*iv*) microscopic examination and (*v*) use of an identification dye, are presented and the results are summarised. Simplified tables show effect of heat and concn. of chemical reagents, longitudinal and cross-sectional appearances of the fibres, colours produced with Texchrome (identification dye) and the odours, residue and behaviour of the fibres when subjected to a flame. E. S. LANE

1573. **Chemical requirements for textiles treated by certain preservative processes.** British Standards Institution (B.S. 2087:1954, 27 pp.).—Methods used by the Government for preserving textiles from fungi, bacteria, insects, weathering and light are described. The type and amount of reagent and its method of application are specified for proofing with Zn naphthenate, Cu naphthenate, Cu-NH_3 (2 processes), mineral khaki (Cr-Fe), Cr-Cu, Cu-Fe, salicylanilide, Cr (chrome tinting), pentachlorophenyl laurate, 2:4-dinitro-1-naphthol and 2-methyl-4:6-dinitrophenol. Appendices deal with the determinations of water-sol. impurities, Cu in cuprammonium proofings, Zn, Fe, Cr and Cu, wax or fat, salicylanilide, pentachlorophenyl laurate, and 2:4-dinitro-1-naphthol and 2-methyl-4:6-dinitrophenol. A. M. SPRATT

1574. Examination of the practical efficiency of textiles. II. Wearing tests. H. Böhringer (*Faserforsch. u. Textiltech.*, 1954, **5** [2], 55–59).—All the factors relating to the problem of accurately assessing the practical efficiency of textiles and their resistance to wear and tear are considered. In addition to the usual physical tests, methods of production, processing and finishing of the materials must be taken into account, and a comprehensive series of tests in use is described and methods are shown of classifying and tabulating these to provide a comparative numerical value for the efficiency in use of the sample or piece of clothing.

M. TADMAN

1575. Use of adsorption columns in the analysis of soap and detergent-stabilised emulsions. R. P. Harker, J. M. Heaps and J. L. Horner (*Nature*, 1954, **173**, 634–635).—The components of aq. emulsions are separated by passage through a column containing Zeokarb 225 mixed with animal charcoal, followed by elution with a series of solvents. The charcoal diminishes the rate of percolation and increases the extent of adsorption, but its role may be purely mechanical. The effluent is free from org. matter; fatty acids (from soap solutions) may be recovered in 98 to 100 per cent. yield by elution with ethanol; lanolin is recovered (99 per cent.) by elution with trichloroethylene; and Na cetyl sulphate is recovered (100 per cent.) by use of ethylene glycol. These figures apply to emulsions of lanolin or vegetable or mineral oils stabilised with long-chain electrolytes.

H. P. PAGET

1576. Determination of methyl groups in polyethylene by infra-red spectrography. E. Borello and C. Mussa (*Gaz. Chim. Ital.*, 1954, **84** [1], 152–156).—At 891 cm^{-1} the difference between total and background optical densities, $\triangle D$, is $1{\cdot}4x + 17y$, where $x = CH_3{\cdot}/C$ per cent. and $y = RR'C{:}CH_2/C$ per cent. for adsorption of light by solid polyethylene (the ratios are in terms of number of C atoms in the groups and the whole molecule). There are absorption bands at 543, 560 and 595 cm^{-1}, due only to $CH_3{\cdot}$, for which $\triangle D = 0{\cdot}26x$, $0{\cdot}28x$ and $0{\cdot}15x$, respectively.

R. C. MURRAY

1577. Methods of testing raw rubber and unvulcanised compounded rubber. II. Methods of chemical analysis. British Standards Institution (B.S. 1673: Part 2: 1954, 46 pp.).—This revision of part of the subject matter of B.S. 902 (Methods of Testing Latex, Raw Rubber and Unvulcanised Compounded Rubber, 1940) includes tests of moisture, acetone extract, acid value, N, total S (3 methods), extractable S (3 methods), ash, Mn (after wet oxidation), Cu and Fe. New tests include alcoholic KOH extraction, direct and difference estimations of rubber hydrocarbon, determination of insoluble matter and dirt and the determination of Sb (after wet oxidation).

A. M. SPRATT

1578. Determination of yellow colour in raw rubber latex films and crepes. G. A. Kidder (*Anal. Chem.*, 1954, **26** [2], 311–315).—A quant. method is described for determining the amount of natural yellow colouring matter in a thin sample of dry rubber or latex film; it is based upon the spectrophotometric examination of the acetone extract of the rubber. The absorptivities of this acetone soln. at 360, 440 and 550 mμ are combined by an empirical equation to give a value for the colour index. Darkening of the rubber or film by oxidative processes does not interfere with the estimation of the yellow colour. Some applications of the method are discussed.

D. BAILEY

1579. Rapid determination of black wattle tannins in spent tannery liquors. D. G. Roux (*J. Soc. Leath. Tr. Chem.*, 1953, **37** [12], 404–407).—The method depends on the intense blue-violet colour produced by Mitchell's ferrous tartrate reagent with the *o*-hydroxyphenolic groups of the tannins in presence of a buffer; colorimetric comparison with a standard soln. is made in Nessler tubes. Twenty to thirty p.p.m. of tannin are detectable and accuracy is within 5 per cent. An approx. spot test is also described, the colour-gradations corresponding with 0·1 to 2·5 per cent. of tannin being listed.

M. TADMAN

1580. Report of the Committee for determination of copper and iron in vegetable tanning extracts. G. Forsyth (Convener) (*J. Soc. Leath. Tr. Chem.*, 1954, **38** [1], 8–10).—The following colorimetric method for determining Fe is submitted for acceptance as a Provisional Official Method of the Society of Leather Trades' Chemists. In preparing the soln., the vegetable tanning extract (5 g of solid or 10 g of liquid) is reduced to ash in a platinum dish at $<500°$ C. The ash is freed from carbonaceous particles without excessive heating either by moistening with a few drops of distilled H_2O, drying and reheating, or by warming with about 10 ml of distilled H_2O, filtering the aq. soln., washing the residue and igniting the filter-paper and residue at low temp. in a platinum dish. The ash is treated with 1 ml of dil. Fe-free HCl (1 + 1) and evaporated to dryness on a water-bath. The residue is redissolved in 2 to 3 ml of dil. HCl (1 + 1) and transferred to a small beaker by means of 25 to 30 ml of distilled H_2O. The soln. is heated just to boiling and the Fe (and any alumina) is precipitated by adding 5 to 6 ml of dil. aq. NH_3 (1 + 1) and keeping the liquid warm for 1 to 1½ hr. The precipitate of iron hydroxide is filtered and washed with warm distilled H_2O containing a little NH_3. The filtrate is saved for Cu determination. Before determining the iron, a standard iron soln. is made by dissolving 0·7022 g of ferrous ammonium sulphate in H_2O and evaporating to dryness on a water-bath with 1 to 2 ml of Fe-free HNO_3, then redissolving in 10 ml of dil. HCl (1 + 1) and making up to 1000 ml. This is diluted ten times as required (1 ml = 0·00001 g of Fe). The precipitate of ferric hydroxide is redissolved by pouring 5 ml of warm dil. A.R. iron-free HCl (1 + 1) on to the filter-paper and thoroughly washing with hot distilled H_2O. The filtrate is cooled and made up to 100 ml (occasionally to 200 ml) with distilled H_2O. A 10-ml aliquot is measured into a 50-ml Nessler tube, diluted with H_2O and 5 ml of dil. HCl (1 + 1) and 5 ml of 40 per cent. KCNS soln. are added. The vol. is made up to 50-ml with distilled H_2O. The colour is matched against a similar Nessler tube containing 5 ml of dil. HCl (1 + 1), 5 ml of 40 per cent. KCNS and H_2O almost to the 50-ml mark, standard iron soln. being carefully added until the tints are identical.

O. M. WHITTON

1581. Examination of the method of Mezger, Rall and Hess for determining the age of writings by means of the chloride test. G. M. Bianca (*Monit. Farm.*, 1954, **60**, 61–64).—The age of writings cannot be estimated from the degree of migration of Cl′ as suggested by Mezger (*Brit. Abstr. B*, 1931, 1061) as too many factors influence the velocity of migration; storage at low humidity inhibits migration and addition of H_2SO_4 to inks accelerates migration.

L. A. O'NEILL

See also Abstract 1496.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1582. **Colorimetric determination of blood calcium with chloranilic acid.** A. E. Teeri (*Chemist Analyst*, 1954, **43** [1], 18–21).—The colour reaction between chloranilic acid and Ca^{++} is used to determine the latter in blood. Mix 2 to 3 ml of serum, 2 ml of H_2O and 1 ml of saturated aq. ammonium oxalate in a 15-ml graduated centrifuge tube. Allow the Ca oxalate to ppt. overnight or at 60° C for 15 min. Centrifuge, drain and wash with 3 ml of H_2O. Suspend the ppt. in 20 ml of H_2O, using successive small portions. Add 10 ml. of aq. 0·1 per cent. chloranilic acid, mix, allow to stand for 4 hr., dilute to vol. and filter. Measure the optical density at 540 mμ. A standard graph is prepared by treating a suspension of Ca oxalate similarly.
G. B. THACKRAY

1583. **Direct determination of cholesterol in serum.** O. S. Bey and A. Leibman (*Bol. Tec. Thermotron*, 1953, **1** [2], 2–3).—The usual solvent extraction and separation is avoided by taking 0·1 ml of serum in 3 ml of glacial acetic acid and adding 2 ml of $FeCl_3$ soln. in conc. H_2SO_4. The colour produced, which is stable for several hours and obeys Beer's law, can be measured on a colorimeter or spectrophotometer. The absorption maximum is about 560 mμ and the error produced by bilirubin and proteins is negligible.
H. PRITCHARD

1584. **Rapid procedure for determination of free serum cholesterol.** H. H. Brown, A. Zlatkis, B. Zak and A. J. Boyle (*Anal. Chem.*, 1954, **26** [2], 397–399).—Two rapid procedures are outlined for the determination of free serum cholesterol. In the first method, 2·5 ml of a filtered soln. containing 2·0 ml of serum in 50 ml of equal parts of ethanol and acetone are treated with 2 ml of a 0·5 per cent. ethanolic (50 per cent.) digitonin soln., 1 drop each of 5 per cent. aq. $AlCl_3$ and conc. aq. NH_3. After mixing and separating by a centrifuge, the residue is heated with 4 ml of 50 per cent. aq. ethanol and 1 drop of 6 *N* HCl. The undissolved matter, cholesteryl digitonide, is separated by centrifugation, dissolved in 3 ml of glacial acetic acid and treated with 2 ml of colour reagent (1 ml of a $FeCl_3$ soln. containing 1 g of $FeCl_3.6H_2O$ in 10 ml of acetic acid is diluted to 100 ml with conc. H_2SO_4). The absorption of the soln. is measured and the cholesterol content of the serum is calculated from a calibration curve. In the alternative method, the 2·5 ml of serum extract are treated with 2 drops of 30 per cent. aq. $AlCl_3$ and 1 ml of 1 per cent. ethanolic (50 per cent.) digitonin solution. The resulting ppt. is separated, washed with 3 ml of acetone, dissolved in 3 ml of glacial acetic acid and treated with colour reagent as before. The two methods compare favourably and are more rapid and sensitive than existing methods.
D. BAILEY

1585. **The micro-estimation of adrenaline in blood by chemical methods.** A. D. Bone (*J. Med. Lab. Technol.*, 1953, **11** [4], 212–223).—The experiments leading to the fluorimetric method of Weil-Malherbe and Bone (*Brit. Abstr. C*, 1952, **306**) are described. Reactions studied include the oxidation of adrenaline to the fluorescent compound adrenolutine, the reaction of adrenaline with ethylenediamine to form a stable condensation product, and the adsorption of adrenaline on acid-washed alumina and its application to blood estimations. An apparatus designed for the simultaneous filtration of up to 8 samples by means of positive pressure from a cylinder of nitrogen is described. Each adsorption unit contains a simple non-return valve consisting of a vertical sintered-glass gas filter (porosity 2), with a layer of mercury on the upper surface of the filter.
F. W. DIGGINS

1586. **A comparison of the determination of gentisic acid in blood by means of the ferric ion - gentisate complex with and without the presence of ferrous ions.** M. Cass and J. G. Wagner (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [1], 50–52).—The method of Gerald and Kagan ($FeCl_2$ - $FeCl_3$ reagent and measurement of the optical density at 595 mμ) and the method of Wagner (use of a reagent containing 1 per cent. w/v of $Fe(NO_3)_3.9H_2O$ in 1 per cent. HNO_3 and measurement at 410 to 420 mμ) for the determination of gentisic acid in tungstate filtrates of bovine blood have been compared and the results examined statistically. Almost identical results were obtained by the two methods over the concn. range 0·125 to 1·75 mg per ml of blood. The method of Wagner is preferable as the reagent and the colour produced are more stable.
N. M. WALLER

1587. **A method for estimating serum acid phosphatase of prostatic origin.** W. H. Fishman and F. Lerner (*J. Biol. Chem.*, 1953, **200** [1], 89–97).—A spectrophotometric method is described for determination of the extent of inhibition of serum acid phosphatase by L-tartrate. It is based on the method of Benotti *et al.* (*Brit. Abstr. C*, 1946, 183) and depends on the extent of hydrolysis of Na phenylphosphate in absence or presence of L-tartrate. The difference in the amount of phenol liberated is believed to represent the inhibition of acid phosphatase of prostatic origin. The method should be useful in the differential diagnosis of cancer of the prostate in men who have normal serum acid phosphatase values.
J. N. ASHLEY

1588. **Clinical tests for ketonuria.** John Nash, John Lister and D. H. Vobes (*Lancet*, 1954, **i** [16], 801–804).—A comparison is made of the sensitivities of the Rothera, Gerhardt and nitroprusside-tablet methods for urinary ketones. The recommended techniques are as follows. *Rothera's test*—Saturate 5 ml of fresh urine with $(NH_4)_2SO_4$ and add 10 drops of 2 per cent. Na nitroprusside soln. Mix and add 10 drops of conc. aq. NH_3, sp. gr. 0·880, and allow the purple colour to develop for 15 min. *Gerhardt's test*—Add 10 per cent. $FeCl_3$ soln. drop by drop to about 2 ml of fresh urine until any ppt. of phosphate redissolves. If reddish-brown, boil 20 ml of urine in an open beaker for 15 min., cool, make up to 20 ml and repeat the test. If a similar colour is given by the boiled urine, it is due to substances other than acetoacetic acid. *Nitroprusside tablet test*—The tablet contains Na nitroprusside, glycine, Na_2HPO_4 and lactose. Place a tablet on a clean white surface and allow 1 drop of urine to fall gently upon it. A purple colour after 30 sec. indicates acetone. Freshly voided urine of ketotic patients was found to contain 9 times as much aceto-acetic acid as acetone. The extreme sensitivity of Rothera's test makes it less useful in distinguishing clinical ketosis than Gerhardt's test, but the latter is subject to much interference by drugs in common use. The tablet test by reason of its speed, moderate sensitivity, convenience and non-interference by

excretory products, drugs or blood is recommended for detecting clinical ketosis.

H. F. W. KIRKPATRICK

1589. **The chromatographic separation of reducing sugar in urine.** R. Williams (*J. Med. Lab. Technol.*, 1954, **12** [1], 43–45).—An ascending technique on triangular shaped Whatman No. 1 filter-paper gives complete separation of reducing sugars within 10 hr. and requires a solvent front run of only 18 cm. The sugars when developed with benzidine appear as brown arcs spreading from edge to edge of the paper. The solvent consists of 3 vol. of *n*-butanol, 2 vol. of pyridine and 1·5 vol. of distilled water. To prepare the developer, dissolve a knifepoint of benzidine in 2 to 3 ml of glacial acetic acid and make up to 20 ml with pure ethanol. The filter-paper hangs with the apex downwards and dipping into the solvent mixture. Five μl amounts are applied near the apex in a continuous line from one side of the triangle to the other. After 10 hr. the paper is dried, rapidly immersed in the developer and dried again. It is then placed in a hot air oven at 100° C for 10 min. The optimum concn. of reducing sugar is approx. 1 per cent. A control soln. is run on another paper at the same time F. W. DIGGINS

1590. **A colorimetric micro-method for determination of glucose.** B. Mendel, A. Kemp and D. K. Myers (*Biochem. J.*, 1954, **56** [4], 639–646).—A colorimetric method for micro-determination of glucose is described. It depends on formation of a bluish-pink colour when dil. aq. glucose is heated with 96 per cent. H_2SO_4; the intensity of the colour is proportional to the concn. of glucose. The aq. glucose (1 ml) containing $\geqslant$15 mg of glucose per 100 ml is added to 96 per cent. H_2SO_4 (3 ml) in a wide test tube, the contents being mixed immediately. The tube is heated in boiling water for 6½ min.; it is then cooled and the intensity of the colour is determined spectrophotometrically at 520 mμ. The colour is stable at room temp. for several hr. For determination in blood, the blood (0·1 ml) is added to aq. trichloroacetic acid - Ag_2SO_4 (1·9 ml) (5 per cent. aq. trichloroacetic acid containing 0·1 per cent. of Ag_2SO_4). After mixing, the suspension is centrifuged at 3000 r.p.m. for 5 min. The clear supernatant liquid (1 ml) is added to H_2SO_4 (3 ml), and the determination is carried out as described above. The reaction appears to be specific for glucose and fructose and saccharides containing these two hexoses. J. N. ASHLEY

1591. **A study of the chromotropic acid reaction for the determination of formaldehyde and its application to the determination of glycine in urine by means of ninhydrin.** D. C. Smith (*J. Med. Lab. Technol.*, 1953, **11** [4], 205–211).—In the procedure described by Daughaday *et al.* (*J. Clin. Endocrin.*, 1948, **8**, 166), the chromotropic acid must be pure and fresh. The colour, which is measured at 565 mμ, is stable for at least 24 hr. When the formaldehyde is produced as the result of a chemical reaction, it is essential to distil it off before applying the colorimetric procedure. For 100 per cent. recovery of formaldehyde, it is necessary to evaporate to dryness, but this may cause errors. Reproducibility is obtained if 60 per cent. of the total possible distillate is collected. The method of Alexander *et al.* (*J. Biol. Chem.*, 1945, **160**, 51) for estimating glycine has been modified. Into a 100-ml round-bottomed flask measure a vol. of the test soln. containing up to 150 μg of glycine, 2 ml of phosphate buffer soln. pH 6·49 and 5 ml of 1 per cent. w/v ninhydrin soln. Make up to 10 ml with water and connect the flask to a water-cooled condenser. Distil into 1 ml of 1 per cent. w/v Na_2SO_3 soln. in a 10-ml conical centrifuge tube graduated in 0·1 ml until the total vol. is 6 ml. Formaldehyde is determined in 3 ml of distillate as before. Values are calculated from a graph made by similarly estimating known amounts of glycine. Both 0·5 and 1·0-ml samples of urine should be used. Recoveries were 98 per cent. when 50 μg of glycine were added to 0·5 ml of urine. F. W. DIGGINS

1592. **Detection and identification of some metabolites of *iso*nicotinic acid hydrazide (isoniazid) in human urine.** W. F. J. Cuthbertson, D. M. Ireland and W. Wolff (*Biochem. J.*, 1953, **55** [4], 669–671).—Methods are described for detecting and identifying certain deriv. of pyridine, in particular deriv. of pyridine-4-carboxylic acid, in urine. The sample of urine (pH 6 to 7) is freeze dried; the residue is suspended in water and the supernatant liquid is applied to filter-paper. After development with *n*-butanol - water or *n*-propanol - water, the compounds on the chromatograms are detected and identified by treatment with CNBr and methylphenylpyrazolone or benzidine, or with picryl chloride. The results show that *iso*nicotinoylglycine and *iso*nicotinic acid are excreted in human urine after therapy with isoniazid. Large amounts of unchanged hydrazide are not excreted.

J. N. ASHLEY

1593. **A hydroxyproline method of analysis for a modified gelatin in plasma and urine.** C. J. Rogers, J. R. Kimmel, M. E. Hutchin and H. A. Harper (*J. Biol. Chem.*, 1954, **206** [2], 553–559).—Because of the large amounts of hydroxyproline present in gelatin and its absence from normal plasma or urine, the determination of the amino-acid is used to measure the content in plasma and urine of a modified gelatin (oxypolygelatin). The method is based on the specific colour reaction of hydroxyproline with ninhydrin. The sample is hydrolysed with aq. HCl in sealed tubes at 145° C for 90 min. After evaporation *in vacuo*, the residue is dissolved in citrate buffer (pH 3·30) and the soln. is passed down a column of Dowex 50 (a sulphonated polystyrene resin); the amino-acid is eluted with citrate, and, after adjusting the pH to 7·0, the ninhydrin reaction is carried out in presence of benzene, and the optical density of the soln. is determined spectrophotometrically at 570 mμ. The mean accuracies of the method for plasma and urine are 99·61 and 95·15 per cent., respectively; the precisions are 100 $\pm$ 5·09 per cent. and 100 $\pm$ 3·24 per cent., respectively. J. N. ASHLEY

1594. **The quantitative determination of hippuric acid.** G. W. Gaffney, K. Schreier, N. Di Ferrante and K. I. Altman (*J. Biol. Chem.*, 1954, **206** [2], 695–698).— A soln. containing hippuric acid is applied to strips of filter-paper, which are then dried in air and sprayed with a soln. of *p*-dimethylaminobenzaldehyde (4 per cent.) in acetic anhydride containing a few crystals of Na acetate, and kept at 130° to 150° C for 1 to 2 min. to form the orange coloured azlactone. This is extracted from the paper with cold methanol, and the intensity of the colour is determined (immediately) spectrophotometrically at 460 mμ. For determination in urine, if sufficient hippuric acid is present, the urine may be applied directly to the paper. Otherwise it is extracted continuously with ethyl acetate for 2 hr.; the extract is then evaporated and the residue is dissolved in ethanol, which is then applied to paper.

J. N. ASHLEY

1595. **Bile acids and steroids. VI. Separation of conjugated bile acids by partition chromatography.** A. Norman (*Acta Chem. Scand.*, 1953, **7** [10], 1413–1419).—The taurine and glycine conjugates of cholic, deoxycholic, lithocholic and cholanic acids are separated by means of reversed-phase partition chromatography on kieselguhr. The moving phase is water or aq. methanol and the stationary phase *n*-butanol, heptane, chloroform - heptane or chloroform - *iso*octanol. The taurine conjugates are quickly eluted from the columns owing to their hydrophilic character. The most suitable solvent systems for different separations are given with diagrams. C. E. SEARLE

1596. **Paper chromatography for the separation of neutral 17-ketosteroids in urine.** S. McDonough (*Nature*, 1954, **173**, 645–646).—Acid hydrolysed urine is extracted with benzene, and the extract is treated with NaOH and dried; it is then concentrated and spotted on Whatman No. 31 filter-paper. The spots should contain <400 μg of 17-ketosteroids. The solvent system used is *cyclo*hexane with 20 per cent. methanol, and the chromatographic tanks are kept at 35° C in a thermostatic cabinet with good air-circulation. The chromatogram is run for 2 hr. by the descending technique, and, after drying, is sprayed with an alcoholic soln. containing 7 per cent. of KOH and 1 per cent. of *m*-dinitrobenzene and heated in a stream of warm air for 10 min. Six ketosteroid fractions have been separated in this manner from male urine, of which three have been identified: (*i*) 11-hydroxy-aetiocholan-3α-ol-17-one, (*ii*) 11-oxo-aetiocholan-3α-ol-17-one and (*iii*) a mixture of aetiocholan-3α-ol-17-one, androstenone and, possibly, 3β-chloro-androst-5-en-17-one. The R_F values range from 0·02 to 0·93. H. P. PAGET

1597. **The separation and identification of mixtures of C_{21} and C_{19} steroids by paper chromatography.** L. R. Axelrod with M. Goldstein (*J. Biol. Chem.*, 1953, **205** [1], 173–184).—A method for paper chromatographic separation of $C_{21}O_3$, $C_{21}O_2$ and $C_{19}O_2$ steroids in a single mixture or as separate groups is described. The solvent systems Decalin - formamide, methyl*cyclo*hexane - propylene glycol, *cyclo*hexene - formamide and methyl*cyclo*hexane - butane-1:3-diol are used in developing the chromatograms. Hydriodic acid, fuming H_2SO_4, $SbCl_3$, *m*-dinitrobenzene, 2:4-dinitrophenylhydrazine and triphenyltetrazolium chloride are used for the detection of the steroids on chromatograms. Absorption spectra of the steroids treated with H_2SO_4 are given as aids in the final identification. The method is applicable to the analysis of blood, urine and tissue homogenates. J. N. ASHLEY

1598. **Hydrolysis of conjugates of urinary corticoids with β-glucuronidase. II. The isolation and determination of tetrahydrocortisone.** B. Baggett, R. A. Kinsella, jun., and E. A. Doisy (*J. Biol. Chem.*, 1953, **203** [2], 1013–1022).—The hydrolysates are subjected to paper chromatography in the propylene glycol - toluene system of Burton *et al.* (*J. Biol. Chem.*, 1951, **188**, 763). After development, the strips are dried and treated with aq. NH_3 - Ag_2O according to the above method. After eluting with methanol, the tetrahydrocortisone is determined by the phenylhydrazine method of Porter and Silber (*Brit. Abstr. C*, 1951, 215). The results show that tetrahydrocortisone is the main α-ketolic steroid extractable from human urine after hydrolysis with bacterial β-glucuronidase. J. N. ASHLEY

1599. **The quantitative determination of amino nitrogen.** F. Bode (*Experientia*, 1953, **9** [7], 271–272).—The reaction of the α-amino carboxylic acids with ninhydrin and $Cu(NO_3)_2$ is used. The resulting red colour is measured spectrophotometrically at 510 mμ (*cf.* Bode *et al.*, *Brit. Abstr. C*, 1953, 214). The amino-acids in molar concn. at pH 6·5 are heated with 1 per cent. ninhydrin soln. in 50 per cent. ethanol. The blue ninhydrin compound is then converted to the red compound with $Cu(NO_3)_2$. When applied to urine, interfering substances (*e.g.*, urea) must be removed by chromatographic separation. N. E.

1600. **Paper-partition chromatography of amino-acids.** P. N. Wahi and R. G. Nigam (*Indian J. Med. Res.*, 1953, **41** [4], 461–465).—R_F values of 34 amino-acids are reported for the one-dimensional descending paper-chromatographic method of Dent (*Brit. Abstr. C*, 1949, 137), in water-saturated phenol as solvent. S. C. JOLLY

1601. **Determination of lysine, tyrosine and arginine.** J. P. Zalta and Y. Khouvine (*Bull. Soc. Chim. Biol.*, 1953, **35** [7], 697–701).—Lysine is determined by reaction with chloramine-T at about pH 6·5; the blue colour formed on heating with Folin - Ciocalteu's molybdotungstophosphoric acid reagent is measured. Amounts from 7 to 30 μg can be determined with a precision of $\simeq \pm 5$ per cent. For tyrosine the reaction with 1-nitroso-2-naphthol in a nitric acid medium is used. On heating, a crimson colour is formed. Amounts from 5 to 30 μg can be determined with a precision of ±3 per cent. For arginine the colour formed in alkaline soln. by 1-naphthol and sodium hypobromite is used; urethane is added to prevent the destruction of the colour by excess of hypobromite. Amounts from 5 to 20 μg of arginine can be measured with a precision of ±3 per cent. N. E.

1602. **Sources of error in microbiological determinations of amino-acids on acid hydrolysates. I. Effect of humin on amino-acid values.** M. J. Horn, A. E. Blum, C. E. F. Gersdorff and H. W. Warren (*J. Biol. Chem.*, 1953, **203** [2], 907–913).—An active material, which increases growth of *Leuconostoc mesenteroides* and *Strep. faecalis*, occurs in acid hydrolysates of cereals and legumes. This material, which appears to be associated with the humin, is formed in most standard methods of hydrolysis, and must be removed by filtering the hydrolysate at pH 4 through sintered glass in order to obtain correct amino-acid values. For limiting amounts of arginine, *iso*leucine, lysine and valine, the effect of the humin is large with media that contain low or high concn. of pyridoxamine. For limiting amounts of histidine and methionine and low concn. of pyridoxamine, the effect of the humin is small; the effect is greater at higher concn. of the vitamin. For limiting amounts of leucine, the humin has no effect with high or low concn. of pyridoxamine. For phenylalanine there is no effect at low concn., but a distinct effect is observed at high concn. of pyridoxamine. J. N. ASHLEY

1603. **Assay of L-phenylalanine as phenylethylamine after enzymatic decarboxylation; application to isotopic studies.** S. Udenfriend and J. R. Cooper (*J. Biol. Chem.*, 1953, **203** [2], 953–960).—A method for the assay of L-phenylalanine in tissues and protein hydrolysates is given. The amino-acid is converted into phenylethylamine by treatment with crude *Streptococcus faecalis* decarboxylase, and the

amine, after extraction into $CHCl_3$, is determined by a modification of the methyl orange procedure (Brodie *et al.*, *Brit. Abstr. C*, **1945**, 276) for org. bases. D-Phenylalanine is not decarboxylated by the enzyme prep., and the method is more specific than either the microbiological assay or the nitration method. The procedure is applicable to radioactive L-phenylalanine. J. N. ASHLEY

1604. **[Determination of] tissue ergothioneine.** D. B. Melville, W. H. Horner and R. Lubschez (*J. Biol. Chem.*, 1954, **206** [1], 221–228).—A chromatographic method for determination of ergothioneine in animal tissues is described. It is similar to that used by Melville and Horner (*J. Biol. Chem.*, 1953, **202**, 187) for the separation of small amounts of ergothioneine from chicken blood, but with modifications, which increase the yield, increase the sharpness of elution bands and decrease the time needed for analysis. The tissue is homogenised with aq. trichloroacetic acid, and the centrifuged homogenate is passed through a column of the acetate form of Amberlite IRA-410. The ergothioneine is eluted by a solvent containing 1 vol. of 98 to 100 per cent. formic acid to 100 vol. of aq. ethanol (75 per cent. v/v). The ergothioneine is determined by means of the magenta colour produced with diazotised sulphanilic acid in presence of alkali as described by Melville and Lubschez (*Anal. Abstr.*, 1954, **1** [2], 329). Blood is treated similarly, except that glutathione and $Na_2S_2O_4$ are added to the laked blood before treatment with trichloroacetic acid. The sensitivity of the chromatographic method is $\simeq 10$ times that of the blood methods. The method is used to show that ergothioneine is of widespread occurrence in tissues of rats fed on stock diets. When purified diets that contain casein as source of protein are used, the ergothioneine content of the blood and tissues is reduced to such low levels as to be undetected by the described method. J. N. ASHLEY

1605. **Electrophoretic separation of proteins on paper and their automatic photometric evaluation.** W. Kemula and W. Bartosiewicz (*Roczn. Chem.*, 1954, **28** [1], 100–108).—The use of electrophoresis on paper for the separation and identification of blood serum proteins is reviewed. Recently Skarzynski, Ostrowski and Mikucki have produced absorption curves on sensitised paper, with the aid of a recording photo-absorptiometer, from mechanically propelled paper chromatograms. It was thus possible to determine, in a mixture of proteins, the percentage of the particular fractions (*Polski Tygodnik Lekarski*, 1952, **7**, 121 and 657). It appears, however, that the absorption curves produced in this way are deformed by oscillations caused by the grain of the chromatographic paper. In this article an improved arrangement is described, which automatically registers well-defined photometric curves. The method has successfully been used for the quantitative evaluation of the proteins in blood serum. The electrophoretic apparatus consists of an aquarium glass, containing six paper strips horizontally placed on two glass rods. The ends of the strips are immersed in two glass troughs placed inside the aquarium, each containing 1 litre of a Veronal - Medinal buffer soln. A 120 to 500 V d.c., max. load 100 mA, is supplied by 2 graphite electrodes immersed in the buffer solutions. An ordinary optical lantern with a selenium photo element in place of the objective lens serves as a photometer. An electrically driven mechanism moves the paper strip in front of a 1-mm wide slot in a metal screen; the variations of voltage of the selenium cell are registered as curves by a polarograph. The particular protein fractions are planimetrically evaluated. Ten to twelve hr. are required for complete separation of the proteins in 0·05 ml of blood serum placed on a 32-mm wide Whatman filter-paper strip. Normal human blood serum was found to contain 7·2 per cent. of total protein, consisting of 54·2 per cent. albumin, 4 per cent. α_1-globulin, 9·1 per cent. α_2, 9·7 per cent. β and 23 per cent. γ. Pathological blood serum (multiple myeloma) contained 6·8 per cent. of total protein, consisting of 27·7 per cent. albumin, 4·5 per cent. α_1-globulin, 8·8 per cent. α_2, 16·0 per cent. β and 43·0 per cent. $\gamma_1 + \gamma_2$. The analytical procedure and assemblage of the apparatus are fully described, and blood serum curves are reproduced. H. BURSTIN

1606. **Detection of proteins on electrophoresis strips by u.v. absorption.** K. H. Kimbel (*Naturwissenschaften*, 1953, **40**, 200–201).—As little was known about u.v. absorption of proteins, a number of serum-protein fractions in 0·1 per cent. soln. in 0·9 per cent. aq. NaCl at pH 7·4 was examined between 240 and 290 mμ in both Beckman and Unicam spectrophotometers; extinction for 0·1 per cent. soln. at 280 mμ was variable, owing to the presence of aromatic amino-acids, but in the region of 250 to 255 mμ the absorption readings of albumins α_1 and α_2, fibrinogen and γ-globulin were reasonably consistent. Owing to adhering lipoids, the β-globulin fraction absorbs more strongly. It was not possible to extract the lipoids without denaturing the proteins, but extraction on the paper can be effected without influencing the absorption of the protein. Particulars of apparatus and methods are given. P. HAAS

1607. **A polarographic study of Evans [azovan] blue and its combination with plasma proteins.** G. Markus and J. P. Baumberger (*J. Biol. Chem.*, 1954, **206** [1], 59–65).—Azovan blue (Evans blue) is readily reduced to the hydrazo compound at a dropping-mercury electrode, and the half-wave potential shifts to more negative values as the pH increases. Protein-bound (rabbit plasma) azovan blue does not furnish a polarographic wave; hence it provides the basis for a new approach to the study of protein-dye equilibrium. An outline of the method is given. J. N. ASHLEY

1608. **Filter-paper electrophoresis: a biophysical laboratory experiment.** M. Martinette (*J. Chem. Educ.*, 1954, **31** [1], 18–19).—Details for setting up an experiment on electrophoresis of blood serum proteins are described. A filter-paper strip moistened with buffer soln. is spotted centrally with serum containing a trace of bromophenol blue and is then clamped between two glass plates smeared with silicone grease to prevent evaporation. The protruding ends of the strip dip into vessels containing buffer solution (pH 7·5) in which electrodes are immersed. A d.c. voltage of 100 to 220 V is applied for 3 to 15 hr. The paper is then removed, dipped in a 1 per cent. soln. of bromophenol blue in ethanol saturated with $HgCl_2$, washed in dil. acetic acid, dried and passed through NH_3 vapour to develop the protein spots. Albumin, α_1, α_2, β and γ-globulins appear as spots leading away from the central line on the filter-paper. G. HELMS

1609. **Possibilities of error in the determination of very small quantities of lactic acid.** G. Kottmeyer (*Biochem. Z.*, 1953, **324** [2], 160–164).—The studies

of a previous paper (*Brit. Abstr. C*, 1952, 309) are continued. The apparatus is freed from O by a stream of nitrogen, which prevents loss of acetaldehyde by oxidation. The best oxidising agent is $KMnO_4$ in the presence of $MnSO_4$. In the titration, the bisulphite soln. must be added to the I soln. in acid medium, and not *vice versa*. N. E.

1610. **Separation and estimation of chitosamine and chondrosamine in complex hydrolysates.** J. E. Eastoe (*Nature*, 1954, **173**, 540–541).—Chitosamine (glucosamine) and chondrosamine (galactosamine) in hydrolysates containing proteins and carbohydrates are quant. separated by chromatography on a 15-cm column of Dowex 50 and estimated by the Moore and Stein ninhydrin method. The amino sugars are well separated from all the known amino-acids except tryptophan, which is destroyed during acid hydrolysis. Loss of amino sugars during hydrolysis is small. C. E. Searle

1611. **A rapid photometric method for the determination of glycogen.** J. Kahan (*Arch. Biochem. Biophys.*, 1953, **47** [2], 408–418).—The colour reaction with anthrone and sulphuric acid is applied directly to a trichloroacetic acid extract of tissue, thus saving alkaline digestion and the pptn. of glycogen by alcohol. The colour is read at 625 mμ. The range of the method is 2 to 300 μg of glycogen, and the experimental error for a duplicate determination is ±2·6 per cent. N. E.

1612. **A colorimetric micro-method for determination of glycogen in tissues.** A. Kemp and A. J. M. Kits van Heijningen (*Biochem. J.*, 1954, **56** [4], 646–648).—A simple method for micro-determination of glycogen in tissues is described. The tissue is extracted with aq. trichloroacetic acid at 100° C, and the glycogen in the extracts is determined, without prior hydrolysis, by the colorimetric method of Mendel *et al.* (see Abstract 1590 above). Results are accurate for 25 to 75 mg of muscle or 10 mg of liver. A single rat muscle weighing 2 g suffices for 15 to 20 determinations and these can be carried out in ≃ 2 hr. If the tissue is previously extracted with 80 per cent. v/v aq. methanol, this extract contains the glucose, which can be determined separately by the same method.
J. N. Ashley

1613. **The separation of choline esters by paper chromatography.** K.-B. Augustinsson and M. Grahn (*Acta Chem. Scand.*, 1953, **7** [6], 906–912).—A method is described for the separation of choline and its esters on paper chromatograms by means of a solvent mixture of *n*-butanol, ethanol, acetic acid and water (8:2:1:3 by vol.). A mixture of ethylene chlorohydrin, *n*-butanol, acetic acid and water (2:10:1:3 by vol.) can also be used. The compounds are detected by spraying with 0·2 per cent. dipicrylamine in 50 per cent. aq. acetone. R_F values of the choline derivatives and some other substances are listed. Acetylcholine and its carboxylic acid esters can be accurately determined by elution with 10^{-4} *M* HCl and estimation with hydroxylamine and $FeCl_3$. Alternatively the eluted compounds can be determined pharmacologically. In preliminary experiments the method is applied to the analysis of mixtures from the enzymatic hydrolysis of choline esters, but difficulties are met with tissue extracts owing to combination of choline and acetylcholine with proteins.
C. E. Searle

1614. **The separation of the phosphate esters of muscle by paper chromatography.** P. C. Caldwell (*Biochem. J.*, 1953, **55** [3], 458–467).—The method of Hanes and Isherwood (*Brit. Abstr. C*, 1950, 402) for separating phosphate esters by paper chromatography is used quant. in the study of these esters in muscle. The muscle is extracted with aq. trichloroacetic acid, and small amounts of Ca and Mg, which would spoil the chromatograms, are converted into sol. complexes with ethylenediaminetetra-acetic acid. The chromatograms are run at room temp. for 16 hr. in *n*-propanol - aq. NH_3, or for 8 to 10 hr. in *tert.*-butanol - aq. picric acid. Individual values obtained with the method are reliable to within 10 per cent. The results show that unidentified phosphate esters are present in the extracts, and that the pyrophosphate fraction is entirely in the form of adenosine di- and tri-phosphates.
J. N. Ashley

1615. **The estimation of saturated and αβ-unsaturated ketonic compounds in placental extracts.** W. H. Pearlman and E. Cerceo (*J. Biol. Chem.*, 1953, **203** [1], 127–134).—A method is described for determination of μg amounts of *allo*pregnan-3β-ol-20-one and progesterone as their semicarbazones; it is applied to the determination of saturated and αβ-unsaturated ketonic compounds in placental extracts. Full term human placenta is estimated to contain 1 to 1·5 mg of progesterone per kg. *Procedure*—Mix the ketonic steroid (50 to 200 μg) or an aliquot (containing ≮200 μg equiv. of progesterone according to determination of u.v. absorption at 240 mμ) of the ketonic fraction of tissue extract with thiosemicarbazide (10 mg) and acetic acid (0·5 ml). Boil gently for ½ min., cool, treat with aq. NaOH (25 ml; sufficient to neutralise 90 per cent. of the acetic acid) and extract with $CHCl_3$ (3 × 20 ml). Wash the combined extracts successively with 4 per cent. aq. $NaHCO_3$ (15 ml) and H_2O (3 × 5 ml). Dry with Na_2SO_4 and filter the soln., evaporate the $CHCl_3$ and thoroughly dry the residue and dissolve it in ethanol. Determine the optical density at 273 mμ (for *allo*pregnan-3β-ol-20-one) or at 301 mμ (for progesterone).
J. N. Ashley

1616. **Determination of liver fat. Comparative studies of different methods.** J. N. Bixby, A. J. Bosch, C. A. Elvehjem and A. M. Swanson (*J. Agric. Food Chem.*, 1954, **2** [7], 375–377).—Methods of fat extraction with wet or dry ether are discussed, and an extraction technique based on the Röse - Gottlieb and Mojonnier methods was adapted for the determination of fat in fresh liver tissue. Glass fat dishes were used and after the additions of the solvents, the mixture was slowly centrifuged for several min. Results are consistently higher (1·1 to 2·8 per cent.) if compared with those of dry ether extraction methods, *e.g.*, modified A.O.A.C. procedure with Goldfisch's continuous extraction apparatus or a Soxhlet; but if mixed solvents, ether, light petroleum or ethanol are used in the dry extraction, results approximate to those of the Mojonnier method.
J. Sci. Food Agric. Abstr.

1617. **A chromatographic radio-autographic method for study of acetate utilisation in animal tissues.** J. Katz and I. L. Chaikoff (*J. Biol. Chem.*, 1954, **206** [2], 887–900).—A procedure that permits a comprehensive study of the utilisation of the ^{14}C of ^{14}C-acetate in 1 g of rat-liver slices is described. The incorporation of ^{14}C into CO_2, lipids and proteins, as well as into 12 to 15 water-sol.

compounds, can be studied simultaneously. The composition of the water-sol. fraction is investigated by paper chromatography in conjunction with radio-autography following the techniques described by Benson *et al.* (*Brit. Abstr. C*, 1950, 147) after prior removal of most of the inorg. salts by rapid electrolytic de-salting in an apparatus similar to, but smaller than, that described by Consden *et al.* (*Brit. Abstr. C*, 1948, 61). The methods are time-consuming, but they are much more rapid and convenient than those involving column chromatography and can be adapted to much smaller amounts of tissue. J. N. ASHLEY

1618. **A colorimetric micro-method for the estimation of chymotrypsin activity.** H. A. Ravin, P. Bernstein and A. M. Seligman (*J. Biol. Chem.*, 1954, **208** [1], 1–15).—The method depends on determination of the esterolytic activity of the enzyme. It will determine chymotrypsinogen (activated to chymotrypsin) in 0·1 to 1·0 mg of tissue. The enzyme or tissue extract is incubated with N-benzoyl-DL-phenylalanine 2-naphthyl ester, and the liberated 2-naphthol is coupled with tetra-azotised di-*o*-anisidine; the resulting bis-azo dye is extracted into ethyl acetate. The colour density of the soln. is measured with a Klett photo-electric colorimeter, a green filter being used (540 mμ), and the amount of enzyme is ascertained from a standard graph. The method is suitable for the routine assay of large numbers of samples. It is inapplicable to the histochemical demonstration of intracellular chymotrypsin. J. N. ASHLEY

1619. **Colorimetric estimation of β-D-glucuronidase activity with 8-benzoylamino-2-naphthyl β-D-glucuronide.** S. H. Rutenburg and A. M. Seligman (*J. Biol. Chem.*, 1953, **203** [2], 731–741).—The biosynthetic prep. of 8-benzamidonaphth-2-yl β-D-glucuronide and a colorimetric method for determination of β-glucuronidase by the use of this substrate are described. The glucuronide is mixed with the enzyme extract and the buffered soln. is kept at 37° C for 4 hr. The liberated 8-benzamido-2-naphthol is then coupled with tetra-azotised di-*o*-anisidine, and the azo dye is extracted with $CHCl_3$. The colour density is determined on a photo-electric colorimeter and the amount of dye is ascertained from a standard graph. A satisfactory histochemical method for demonstrating β-glucuronidase activity is not provided by the substrate. J. N. ASHLEY

1620. **Turbidimetric estimation of hyaluronidase.** R. H. Pearce (*Biochem. J.*, 1953, **55** [3], 467–472).—Use of the turbidity reaction as a basis for assay of hyaluronidase requires that the kinetics of the reaction and the effects of concn. and mode of prep. of both the substrate (hyaluronate) and enzyme on the apparent enzymic activity be known. These are investigated. The turbidity-reducing activity of hyaluronidase follows first-order kinetics under standard conditions of assay, provided that the time of incubation is >10 min., and the concn. of enzyme is >6 turbidity-reducing units. Under these conditions, the following relation $S_d = a(E - e)S_o^b$ holds, where S_d is the amount of substrate destroyed, S_o is the initial concn. of substrate, E is the concn. of enzyme, and a, b and e are const., the first two being dependent mainly on the protein content and specific viscosity of the substrate, respectively. This formula cannot be made the basis for a determination until uniform preparations become available, but useful comparisons of enzymic activities can be made. J. N. ASHLEY

1621. **Biological assay of hyaluronidase on rabbits.** V. M. Venturi (*Acta Pharmacol. Tox., Kbh.*, 1953, **9**, 93).—Hyaluronidase added as a single separate injection into a subcutaneous site being infused with saline alters the rate at which the NaCl infuses into the subcutaneous tissues. The change in rate is related to the amount injected in a manner suitable for use in assays of hyaluronidase. B. BASIL

1622. **The determination of deoxyribonuclease activity with the optical ultra-centrifuge.** J. G. Rabatin, R. Friedland and W. J. Frajola (*J. Biol. Chem.*, 1953, **203** [1], 23–33).—Deoxyribonuclease activity is quant. studied by means of the optical ultra-centrifuge and a soln. of calf thymus nucleoprotein in *M* NaCl soln. The results show that degradation of the protein by the enzyme involves (*i*) a change in the shape of the protein mol., (*ii*) a specific depolymerisation (or change in size) that produces a small number of like particles and (*iii*) a further cleavage into smaller particles. J. N. ASHLEY

1623. **Studies on sulphatases. V. The determination of inorganic sulphate in the study of sulphatases.** K. S. Dodgson and B. Spencer (*Biochem. J.*, 1953, **55** [3], 436–440).—An adaptation of the benzidine method for determination of small amounts of enzymically liberated H_2SO_4 in presence of acetate-buffered biological material is described. The benzidine sulphate is tetra-azotised and then coupled with alkaline thymol. The intensity of the red colour ($\lambda_{max.}$ 500 mμ) is determined on an absorptiometer, and the amount of SO_4'' is ascertained from a standard graph. By this method K_2SO_4 is recovered quant. from tissue suspensions of different organisms under various conditions of pH and buffer molarity and after incubation at 37·5° C for 1 hr. Except for Ba, recoveries of SO_4'' are unaffected by presence of small amounts of K, Na, Mg, CN', Cl, F, Fe, Ca or PO_4'''. Within certain limits K *p*-acetylphenylsulphate, K glucose 6-sulphate, K myronate and chondroitinsulphuric acid have no effect on the method. J. N. ASHLEY

1624. **A rapid spectrophotometric assay for co-enzyme A.** R. W. von Korff (*J. Biol. Chem.*, 1953, **200** [1], 401–405).—A rapid (15 to 20 min.) and specific method for the spectrophotometric determination of catalytic amounts (0·5 to 1·5 Lipmann units) of co-enzyme A is described. It depends on determination of the rate of reduction of diphosphopyridine nucleotide by α-oxoglutarate catalysed by a sol. oxidase and co-enzyme-A. The latter is continually regenerated from the succinyl co-enzyme-A formed in the reaction by means of a succinyl co-enzyme-A deacylase. J. N. ASHLEY

See also Abstracts 1445, 1446, 1451, 1461, 1491, 1732.

Drugs

1625. **Spectrophotometric determination of morphine and codeine.** W. A. Clark and A. J. McBay (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [1], 39–42).—Two spectrophotometric methods, one absolute and one relative, are described for the determination of codeine and morphine; the absolute method is preferable as no standard is required. Measurements are made over the range 220 to 325 mμ and morphine is distinguished from codeine by the shift of its max. absorption when in strongly alkaline soln. from that in acid soln. Equations are presented for the calculation of the concn. of the

alkaloids in unknown samples. The percentage error for the relative method is from 0·4 to 2·46 per cent., and for the absolute method from 0·85 to 2·34 per cent. N. M. WALLER

1626. **Determination of ephedrine in oily and aqueous nasal sprays.** L. G. Chatten and M. Pernarowski (*Drug Standards*, 1954, **22** [1–2], 1–6).—For oily preparations 10 ml are dissolved in 50 ml $CHCl_3$ and titrated with 0·1 *N* perchloric acid soln. with crystal violet as indicator. The reagent, which is made by dissolving 8·4 ml of 70 to 72 per cent. $HClO_4$ in 200 ml of glacial acetic acid, adding 10 ml of acetic anhydride and diluting to a litre with glacial acetic acid, is standardised against K acid phthalate. Aqueous sprays are made alkaline and the alkaloid is extracted with $CHCl_3$. The ephedrine is titrated directly in the $CHCl_3$ soln. N. E.

1627. **Chromatography of alkaloid reactions.** F. M. Shemyakin, A. W. Karpov and N. K. Medvedeva (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1953, **90** [3], 399–402).—The various standard reactions for alkaloids are much more specific if carried out chromatographically on Al_2O_3 columns or on paper. The reactions of morphine with HNO_3, H_2SO_4 and HNO_3, H_2SO_4 and $(NH_4)_2VO_4$, $FeCl_3$, and ammoniacal cerium nitrate, and of codeine with HNO_3, H_2SO_4, $FeCl_3$, and Frohde's reagent, and various methods of determining both these alkaloids in a mixture are described. R. C. MURRAY

1628. **Fluorimetric determination of digitoxigenin.** K. B. Jensen (*Acta Pharmacol. Tox., Kbh.*, 1953, **9**, 66–74).—When treated with hydrogen peroxide in strong HCl and in the presence of ascorbic acid, digitoxigenin (2 to 20 μg in 10 ml of test soln.) gives a fluorescence proportional to the amount present. Equivalent molecular quantities of digitoxin and purpurea glycoside A give the same intensity; purpurea glycosides of the B series give only a weak fluorescence. The reproducibility is ±5 per cent. W. H. C. SHAW

1629. **Paper chromatography of cardiac glycosides and aglycones from *Digitalis purpurea*.** K. B. Jensen (*Acta Pharmacol. Tox., Kbh.*, 1953, **9**, 99–108).—The substances are separated by one-dimensional filter-paper chromatography with a mobile phase of $CHCl_3$ - water - methanol (5:5:1 by vol.) and on formamide-impregnated filter-paper with a $CHCl_3$ - benzene - formamide mixture as mobile phase. Spot tests for the substances are also described. W. H. C. SHAW

1630. **Paper-chromatographic detection of new glycosides in *Digitalis purpurea*.** K. B. Jensen (*Acta Pharmacol. Tox., Kbh.*, 1953, **9**, 275–290).—Glycoside mixtures from stabilised leaves are analysed by one-dimensional chromatography on formamide-impregnated filter-paper. The presence of five new substances is demonstrated; three of these react to colour reagents like the A series of purpurea glycosides and two like the B series. The proportions of known to unknown glycosides is roughly estimated and various breakdown products, particularly in non-stabilised leaves, are shown to be present. W. H. C. SHAW

1631. **Structure of picrotoxin. I. Relation between picrotoxinin and picrotin in picrotoxin. Identification of picrotoxin.** E. J. Hansen and B. Jerslev (*Dansk Tidsskr. Farm.*, 1954, **28** [2], 25–33).—Comparative X-ray analysis and mixed m.p. determinations confirm the view that picrotoxin (**I**) is an equimol. compound of picrotoxinin (**II**) and picrotin (**III**). The eutectic m.p. of **I** or **II** with phenobarbitone (Danish Pharmacopoeia test) are (like the individual m.p.) almost identical. To distinguish between **I** and **II**, ≃1 mg of the sample is gently heated on a microscope slide with 1 drop of *N* NaOH until the soln. becomes yellow; on acidification **III**, but not **II**, is pptd. as needles having m.p. 248° to 253° C (as determined under the microscope at a heating rate of ≃5° per min.). P. S. ARUP

1632. **The infra-red determination of erythromycin.** W. H. Washburn (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [1], 48–49).—A method for the determination of erythromycin is described that is specific, reproducible within the range ±1 per cent. and rapid (30 min.). A 120-mg sample is dissolved in 4 ml of $CHCl_3$ and the soln. is transferred to a 0·5-mm cell. By use of an i.r. spectrophotometer, slit width 0·42 mm, and a wavelength drive speed of 1 turn per 2 min., the spectrum is scanned from 10·3 to 10·7 μ, and the absorption at 10·46 μ is determined by the base-line method. The percentage purity of the sample is calculated by comparison of this value with a calibration graph. Results agree well with the biological activity of the samples. N. M. WALLER

1633. **The ultra-violet spectrophotometric determination of official phenobarbitone preparations.** L. N. Mattson (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [1], 22–24).—Procedures for the assay of phenobarbitone in U.S.P. tablets and elixir and in capsules with ephedrine sulphate are described. The optical density at 240 mμ of phenobarbitone soln. in borate buffer at pH 9·5 is measured. The results of 21 assays show that there is satisfactory agreement with the U.S.P. extraction method and the method is applicable to tablets containing stearates. N. M. WALLER

1634. **On the assay of aminophylline with phenobarbitone sodium tablets.** S. Bhattacharya and S. C. Banerjee (*J. Instn. Chem.* (India), 1954, **26** [1], 33–37).—The method, said to be 100 per cent. accurate, depends upon the differing solubilities of phenobarbitone silver and the sodium salt of theophylline in aq. ammonia. After digestion of the tablets with dil. NH_3 soln., the filtered extract is warmed with dil. aq. NH_3 and $AgNO_3$ soln. On cooling and filtering, the theophylline in the ppt. and the phenobarbitone in the filtrate can be determined by known methods. M. TADMAN

1635. **A note on the determination of Novalgin.** C. M. P. Wirth (*Drug Standards*, 1953, **22** [1–2], 14–15).—A modification of the iodimetric method for the determination of phenazone is applied to Novalgin (sodium phenyldimethylpyrazolonylmethylaminomethanesulphonate). Novalgin (300 mg) is dissolved in 50 ml of water, 3 ml of dil. acetic acid (6 per cent. v/v) are added and the titration is carried out with 0·1 *N* I, starch being added towards the end of the titration. N. E.

1636. **The *in vitro* evaluation of the antibacterial activity of sulphonamides.** G. M. Naimark and L. White (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [1], 7–8).—Factors that affect the results of *in vitro* antibacterial tests applied to sulphonamide-containing pharmaceuticals are discussed. Sulphonamide inhibitors must be excluded from the media used, and such variables as the composition and pH of the medium, the species and strain of the test organism, the inoculum age, size and diluting fluid,

the concn. and stability of the drug soln., the temp. and duration of incubation and criteria used to determine activity are all of importance. The Strauss modification (*J. Immunol.*, 1941, **42**, 331) of Knight's culture medium appears to be inhibitor free; by use of this medium it is established that the growth of *M. pyogenes* var. *aureus* is inhibited by soln. and ointments containing 30 per cent. of Na sulphacetamide. N. M. WALLER

1637. **Polarography of picolinic and *iso*nicotinic acid and their amides.** H. H. G. Jellinek and J. R. Urwin (*J. Phys. Chem.*, 1954, **58** [2], 168–173).—The polarography of picolinic and *iso*nicotinic acids and their amides is studied over a range of pH values. The wave height decreases rapidly when a certain pH value is reached, but whereas each acid shows only one type of wave, the amides have two different types of waves. The dependence of wave heights on pH is discussed in terms of re-combination reaction taking place in the electrode interface. A. JOBLING

1638. **Microtoxicology. X. Colorimetric reactions and optical crystallographic properties of five synthetic antispasmodics.** T. J. Haley and G. L. Keenan (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [1], 46–48).—The antispasmodics Profenil citrate, Syntropan phosphate, Trasentin hydrochloride, Pavatrine hydrochloride and lachesine chloride were tested with various colorimetric and pptn. reagents, and their m.p. were determined. Although definite differences were observed, the use of their optical crystallographic properties provides the simplest means of identification. N. M. WALLER

1639. **Paper-chromatographic separation of thiouracil derivatives.** F. Reinhardt (*Mikrochim. Acta*, 1954, [2], 219–222).—The paper-chromatographic separation of thiouracil derivatives is described. The solvent used is an (8 + 3) mixture of benzene and ethanol. A procedure is given that takes account of the volatility of the solvent. The thiocarbamide reaction with ruthenium chloride is used in the development of the chromatogram. The reaction occurs in acid medium. In the test-tube reaction, HCl is used, but because of its action on the paper it is replaced by trichloroacetic acid, which, however, makes the test somewhat less sensitive. The method was used for the separation of 6-amino-, 6-methyl- and 6-propyl-thiouracil from each other and from thiourea and mercaptobenziminazoledimethylol (Thyreocordon). A. J. MEE

1640. **Nephelometric determination of bacterial vaccines.** E. K. Narayanan and A. L. Bhatia (*Indian J. Med. Res.*, 1953, **41** [3], 303–306).—The $BaSO_4$ suspensions in Na citrate soln. suggested by Brown (*Ind. J. Med. Res.*, 1914, **1**, 711) as nephelometric standards for assessing the concn. of bacterial vaccines are sufficiently accurate for general use. The opacities of new standards should be checked photo-electrically. S. C. JOLLY

1641. **The precision of biological assays.** D. J Finney (*Acta Pharmacol. Tox., Kbh.*, 1952, **8**, 55).—The precision of randomised block designed assays is better estimated by the intra block-residual error than by interaction of doses and blocks. The latter is an estimate of the former together with a component due to the characteristic slope value of each block. This latter component, being not rightly considered as error, may be eliminated; the statistical analysis of parallel line and slope ratio assays is thereby eliminated. B. BASIL

1642. **The paper electrophoresis of drugs. Changes in direction of travel due to additives.** K. G. Krebs and A. Wankmüller (*Deutsche Apotheker Z.*, 1954, **94** [12], 234–236).—*Anionic substances*—The changes in rate of movement of 7-hydroxy-4-methylcoumarin, scopolin and scopoletin, when the paper is treated with buffers and reagents, is studied. Neither changes in the buffer alone nor in reagents when applied with the spot of the test substance have much effect. Pre-treatment of the paper by soaking in mixtures of buffer and different concn. of reagents markedly changes the rate according to the concn. of added reagent. Myxal and Zephirol solutions (cationic) decrease or reverse direction of travel, Praecutan and Quartamon solutions increase it. *Cationic substances*—Similar effects are observed when succinylcholine chloride, gallamine tri-ethiodide, Megaphen, Padisal, Latibon, phenothiazine and rutin are placed on paper treated with Praecutan soln. *Non-ionic substances*—Although these normally remain where spotted, their behaviour is also modified by treatment of the paper. P. S. STROSS

1643. **Laboratory investigations in suspected industrial poisoning.** R. E. Lane (*Brit. Med. J.*, 1954, [i], 978–980).—This review article covers laboratory tests that are applied in cases of poisoning by lead, mercury, arsenic, carbon monoxide, aromatic amino- and nitro-compounds, trinitrotoluene, benzene, selective weed-killers and organic phosphorus insecticides. N. E.

Food

1644. **Determination of moisture in cereals: review of methods in common use.** A. Bennett and J. R. Hudson (*J. Inst. Brewing*, 1954, **60** [1], 29–34).—This review, with 47 references, covers oven drying, distillation, electrical and chemical (based on the Karl Fischer technique) methods. Whilst the results given by the several methods agree within their respective series and are sufficiently accurate for many commercial purposes, they differ according to the method used. Further investigation is needed in order to reach a definition of true moisture content and an absolute method for its determination. P. S. ARUP

1645. **Study of methods for determination of moisture in malt.** A. Bennett and J. R. Hudson (*J. Inst. Brewing*, 1954, **60** [1], 35–42).—Results for malt obtained by drying at 40° C in a current of N at a pressure of 0·5 mm of mercury, by the Karl Fischer method and by the Dean and Stark distillation method with toluene agree satisfactorily. The substantially lower results obtained by the standard oven-drying method are probably explained by the permanent retention of water owing to enzymatic proteolytic activity; evidence for this view is afforded by the marked change in the course of the drying of normal, but not of black, malt, observed over the temp. range of 74° to 79° C, and also by differential thermal investigations with the use of thermistors. Drying at 40° C in a current of N at 0·5 mm of mercury is probably suitable as a precise reference method. As a routine method, the Karl Fischer method (extraction of the water from the malt by stirring with methanol for 2 hr., and direct titration) is preferable to the distillation method. See also Abstract 1644. P. S. ARUP

1646. **Use of cobaltous chloride to detect moisture patterns in partly dehydrated kernels of corn.** C. B. Ward, jun., and R. G. Tischer (*Cereal Chem.*, 1953, **30**, 420–426).—A soln. of 10 g of crystalline $CoCl_2.6H_2O$ in 5 ml of 99·5 per cent. methanol was used to determine moisture contents ranging from 70 per cent. to 0·5 per cent. in maize grains. Whether the residual moisture was located at the surface or at the centre of the grain depended on the method of drying. The limit of sensitivity to the test was a moisture content of 4 per cent.

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1647. **Rapid method of estimating the activity of β-amylase in barley extracts.** B. H. Kirsop (*J. Inst. Brewing*, 1953, **59**, 378–381).—In a rapid and reliable method for estimating β-amylase, a 1 per cent. soluble starch soln. is hydrolysed for 3 min at 20° C by 1 ml of enzyme extract. Hydrolysis is stopped by heating the mixture for 5 min. at 100° C with the addition of 2 ml of 1 per cent. 3:5-dinitrosalicylic acid in 0·4 *N* NaOH buffered with 30 per cent. Rochelle salt. The reducing sugars from the hydrolysis convert dinitrosalicylic acid into a red-brown derivative whose colour intensity is measured.

J. Sci. Food Agric. Abstr.

1648. **The determination of crude fibre. II.** V. P. Hirsjärvi with L. Andersen (*Z. anal. Chem.*, 1954, **141** [5], 348–361).—The various modifications of the Weender method are reviewed. A further modified method is described; this is similar to that of Puranen and Tomula (*Acta Chem. Fenn.*, 1930, **3**, 85), but a special glass-wool filter is used. Comparisons made with three other modifications on 23 feeding stuffs show that the new method is speedier and has better reproducibility. An extensive bibliography is appended. E. Hayes

1649. **A routine method for estimating the starch content of wheat by-products.** R. A. D'Arcy (*Cereal Chem.*, 1954, **31** [1], 37–42).—A routine method is described for the determination of starch and insol. pentosans in wheat by-products such as bran, pollard (shorts), etc. Two 1·0-g samples are treated for 10 min. with ≃ 50 ml of dil. HCl soln. (2 per cent. v/v plus 0·5 ml of Teepol per litre) with occasional stirring; the suspensions are centrifuged at 2500 r.p.m. for 7 min. and the supernatant liquids are decanted through Whatman No. 41 filter-paper. The residues are extracted twice more, combined, dispersed in 180 ml of water and boiled gently for 2½ hr. with 20 ml of HCl, sp. gr. 1·125. The soln. is cooled, diluted to 250 ml with water and filtered through a Whatman No. 40 filter-paper. The reducing power of a 15-ml aliquot (≡ 120 mg of sample), neutralised to methyl orange with solid Na_2CO_3, is determined by the Schoorl method (Flohil, *Cereal Chem.*, 1933, **10**, 471). To a 150-ml aliquot at 30° C, adjusted to pH 6·0 with 30 per cent. NaOH soln. and finally with 0·1 *N* NaOH, 1·0 g of washed bakers' yeast is added, and the mixture is fermented for 16 hr. at 30° C. After cooling and diluting to 250 ml, the suspension is centrifuged, and the reducing power of a 25-ml aliquot (≡ 120 mg of sample) of the supernatant liquid is determined. The percentage of starch = 0·775 *G* where *G* = mg of glucose corresponding to the difference between the thiosulphate titre before and after fermentation. Approx. percentage of endosperm or flour = 1·12 *G*, assuming that wheat endosperm contains 69·3 per cent. of starch at 14·5 per cent. moisture. The residual sugars after fermentation represent pentoses from insol. pentosans; approx. percentage of insol. pentosans = 0·73 × mg of pentose (obtained from glucose table). The method is accurate to within about ±2 per cent.

S. C. Jolly

1650. **A method for the quantitative determination of albumins and globulins in wheat flour.** J. W. Pence, N. E. Weinstein and D. K. Mecham (*Cereal Chem.*, 1954, **31** [1], 29–37).—A method, in which interference from gliadin is largely eliminated, is described for the determination of albumins and globulins in flour with an accuracy of approx. ±10 per cent. Sol. proteins are extracted by dispersing 40 g of flour in 350 ml of 0·5 *M* NaCl buffered at pH 6·8 with 0·1 per cent. of phosphate, shaking gently for 30 to 40 min., centrifuging and decanting the supernatant liquid; the residue is extracted twice more with 300 to 350 ml of fresh extractant. Total and non-protein N is determined in a 50 or 100-ml aliquot of the combined supernatant soln. The remainder of the extract is dialysed overnight against running tap water followed by two changes of distilled water, concentrated under reduced pressure, freeze-dried and weighed (*W*). Tryptophan N (*N*) in the residue is determined by the colorimetric method of Spies and Chambers (*Brit. Abstr. C*, 1950, 64) and amide N (*M*) by method of Mecham and Olcott (*Ind. Eng. Chem.*, 1947, **39**, 1023). The relative proportions of albumins (*x*), globulins (*y*) and gliadin (*z*) are calculated from the equations—

$$x = 0{\cdot}0023\,M + 0{\cdot}540\,N - 0{\cdot}408$$
$$y = -\,0{\cdot}0547\,M - 0{\cdot}472\,N + 1{\cdot}672$$
$$z = 0{\cdot}0524\,M - 0{\cdot}0683\,N - 0{\cdot}264.$$

The albumin and globulin contents of the flour are obtained by multiplying *x* and *y* by *W*.

S. C. Jolly

1651. **Polarographic investigation of the proteolytic activity of grain and flour enzymes.** G. I. Kotlar (*Przem. Rol. Spoz.*, 1953, **7** [11], 387–390).—The existing methods for the determination of the proteolytic activity of grain and flour enzymes are either too troublesome or they fail to indicate the early stages of proteolysis that are so significant for the baking process. Albumins and their hydrolysates, hence also grain and flour enzymes, owe their proteolytic activity to the presence of polypeptides and amino-acids. A polarographic method for the determination of proteolysis based on earlier research by Shamshikova (1940) and Moskalieva (1948) is described in which the increase of the catalytic albumin wave resulting from the autolysis of aqueous grain extracts, is measured. A mixture of 0·003 *N* $Co(NH_4)_6Cl_3$, 0·1 *N* NH_4Cl and 0·1 *N* aq. NH_3 serves as a buffer soln. (pH 9·5). The grain is milled to 1 mm, and 2-g portions, weighed to 0·01 g, are placed in 50-ml conical flasks, each charged with 20 ml of toluene-saturated distilled water. The samples are shaken until uniform suspensions are obtained and are then put into a thermostat at 20° C. Shaking is repeated every 10 min. and the samples are filtered through dense filter-paper at pre-determined intervals. Five min. after filtration has started a filtrate specimen is tested polarographically. The catalytic wave is best observed when 4·9 mg of the electrolyte is used to every 0·1 mg of grain extract (equivalent to 9·36 mg per litre of water-soluble nitrogen and 4·3 mg per litre of N not precipitable by 2 per cent. trichloroacetic acid). The increase of the catalytic albumin wave in autolytic grain extract agrees within 3 per cent. with the increase of the concn. of water-soluble N and N not precipitable by 2 per

cent. trichloroacetic acid. Both methods indicate the breakdown of grain albumin, but the polarographic test defines more clearly the start of proteolysis. Thus an assessment can be made of the proteolytic activity in overgrown grain and in flour stored over a longer period. Finally, it provides a means to differentiate between particular species and crops of grain and is suitable for routine tests.

H. BURSTIN

1652. **Method of determining enzymic digestion of raw starch.** R. L. Gates and R. M. Sandstedt (*Cereal Chem.*, 1953, **30**, 413–419).—Starch (0·5 g) is digested for 20 hr. in 20 ml of soln. containing buffer, micro-organism inhibitor and enzyme, the enzyme activity being measured by the loss in wt. of the filtered dried undigested residue. With a given bacterial enzyme prep., the amount of digested starch, determined as maltose, was relatively constant (64 to 73 per cent.). As a digestion unit, the enzyme activity required to digest 20 per cent. of 1 mg of raw maize starch in 20 hr. is proposed.

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1653. **The use of microbiological methods in laboratories supervising the production of potato starch.** E. Rzechowska (*Przem. Rol. Spoz.*, 1953, **7** [10], 345–347).—Crude potato starch contains $\simeq$20 per cent. of water, including some cellular fluid that surrounds the starch globules. This contains phosphatides and albumin, which favour the growth of bacteria mostly derived from the water used in the production of starch. Whereas bacteriological examination of drinking and industrial waters is generally accepted, no such regulations exist in respect of starch. The paper presents detailed information on the execution of suitable bacteriological tests and suggestions about bactericidal treatment of water used in the production of starch.

H. BURSTIN

1654. **Determination of invert sugar, cane sugar, fructose and glucose.** F. Wobisch and J. Schneyder (*Öst. Chem.-Ztg.*, 1954, **55**, 40–46).—The Lehmann method for fructose and the Allihn method for glucose are shown to give results incomparable with those by the Meissl method. As tables for the Meissl method have not hitherto been determined over a full range, the requisite analyses have now been made. The Meissl method is described in detail and tables are given of invert sugar, cane sugar, fructose and glucose equivalents of Cu_2O from 10 to 425 mg, with interpolation figures for steps of 0·1 mg of Cu_2O.

SUGAR IND. ABSTR.

1655. **Reagent for the detection of sugar in condensate water.** T. Kozakiewicz (*Gaz. Cukr.*, 1953, **55**, 162–164).—The reaction of ammonium molybdate to give a blue colour with fructose in alkaline soln. or with other sugars in acid soln. has been found to be as sensitive as the 1-naphthol reaction. The reagent is prepared by dissolving 60 to 80 g of ammonium molybdate in 1 litre of conc. H_2SO_4 heated just to boiling. If the reagent gives a blue colour with dist. H_2O, $KMnO_4$ is then added to the initial reagent in amount found sufficient by test to prevent such a "blank" reaction; a slight excess of $KMnO_4$ may be needed if NH_3 is present in the water. The test is carried out by mixing 0·5 ml of reagent with 0·5 ml of the water. With 5 per cent. molybdate in the reagent, 1 part of sugar in 10,000 can be detected; with 10 per cent., 1 part in 100,000 can be traced.

SUGAR IND. ABSTR.

1656. **Chromatographic separation of sugars with hydrocellulose.** J. D. Geerdes, B. A. Lewis, R. Montgomery and F. Smith (*Anal. Chem.*, 1954, **26** [2], 264–266).—A hydrocellulose is prepared by dissolving cellulose powder (5 g) in 85 per cent. H_3PO_4 (250 ml) and pptg. it by pouring into water (3 litres). This hydrocellulose is compared with untreated cellulose with respect to its capacity and resolving power for the separation of sugars and their methyl derivatives. With an ethyl methyl ketone and water mixture as developing solvent, a column containing hydrocellulose and cellulose is superior to one containing only cellulose for the separation of methylated sugars, but there is no significant difference between the two types of adsorbent for the separation of free sugars with *n*-butanol, ethanol and water mixture as solvent. The success of the modified column (40 cm × 3 cm) is attributed, in part, to the top of the column being kept in place by a metal weight. Overloading becomes evident only when 4·5 g of a methylated sugar (2:3:4:6-tetra-O-methyl-D-glucose) is added to the column. Rhamnose ethyl thiol, m.p. 133° to 134° C, when treated with ethanol, $HgCl_2$ and yellow HgO gives a mixture of α- and β-ethyl-L-rhamnofuranosides, $[\alpha]_D^{23}$ — 18·9° (in water). This mixture is separated into the anomeric forms by fractionation on a hydrocellulose - cellulose column giving α-ethyl-L-rhamnofuranoside (m.p. 56·5° to 57·5° C; $[\alpha]_D^{25}$ — 98° in water) and β-ethyl-L-rhamnofuranoside (m.p. 21° to 24° C; $[\alpha]_D^{23}$ + 105° in water). The column is used for the separation of 2:3:4-tri-, 2:3-di-, 2- and 3-O-methyl-D-xylose and D-xylose obtained by hydrolysis of the methylated hemicellulose from the straw of flax and for the separation of the cleavage fragments of methylated aspen-wood hemicellulose.

D. BAILEY

1657. **International commission for uniform methods of sugar analysis. American national committee.** Anon. (*Int. Sugar J.*, 1954, **56**, 11–13).—Some of the reports submitted to the 1953 meeting are summarised, as follows. Subject 2: *Weighing, taring, sampling and classification of sugars.* Procedure at Revere sugar refinery is described in detail and fundamental points for bag handling are indicated. Subject 3: *Ash content of sugar products.* A reference method for reporting conductimetric ash has been established by a sub-committee on methods of the sugar-bottling industry, and good agreement has been obtained in collaborative tests, the spread in ash values being only 0·002 per cent. of ash. Details are given of the method, the C-ratio to be used and of temperature corrections. Subject 7: *Refractive index of solutions of sucrose, dextrose, laevulose, raffinose, invert sugar and mixtures.* The Martin values of 60 to 70 per cent. sucrose solutions have been checked, but the equation computed from observed values is not yet satisfactory. Subject 10: *Evaluation of refining qualities of raw cane sugar.* A provisional simplified wet screening method, without the use of a Wagner shaker, is described. Subject 12: *Evaluation of the crystallising qualities of beet and cane-factory juices.* From a theoretical examination of the induction period, the period of growth proper and the late period, an equation has been produced by applying the activity theory to actual sugar juices: log [velocity (*impure*)/velocity (*pure*)] = log (relative velocity) = iI, where I = concentration of non-sucrose solute in g per 1000 g of water and i is a constant designating the melassigenic factor. The equation has been found valid for a wide variety of

sugar house products. The method of testing is described. Subject 13: *Colour and turbidity of sugar products.* Definitions of terminology for photo-electric measurements, as developed by Deitz *et al.*, are given, including transmittance, transmittancy, absorbancy, absorbancy index and attenuation index. The industrial method developed for colour determination of white sugars is described. Subject 15: *Dry substance in sugar and sugar products.* A review of the Karl Fischer method has been made. The method gives very reproducible results, but does not appear to be more accurate than gravimetric methods.

SUGAR IND. ABSTR.

1658. **Further studies on the hypo-iodite method for the determination of sugars and sugar-like substances.** M. Lüdtke (*Z. anal. Chem.*, 1954, **141** [5], 337–344).—In the determination of uronic acids in hydrolysates of plant materials by the hypo-iodite procedure, hydrolysates of polyuronic acid (pectins and alginic acids) give abnormally high results. The effect is not due to the presence of furfural or to the formation of iodoform; the amount of iodoform formed is small and its formation may be repressed by substituting $NaHCO_3$-Na_2CO_3 for NaOH. The same effect is found with the monomeric acids after these have been boiled with water; the Cu values found are lower than the theoretical. The simple sugars give normal results with the Cu or the hypo-iodite procedure, although discrepancies occur with galactose and rhamnose. Glucosamine hydrochloride gives hypo-iodite values 2·5 times the Cu values even in unboiled soln.; chitose behaves in the same way. E. HAYES

1659 **Serbia's method for determination of unfermentable reducing sugars [in molasses].** F. W. Zerban (*Sugar, N.Y.*, 1954, **49** [2], 40–41).—By use of 80 g of a (1 + 1) mixture of Permutit A and Zeokarb H for 400 ml of 1·6 per cent. w/v molasses soln. and subsequent clearing with neutral Pb acetate soln. (Serbia's method, *cf. Sugar, N.Y.*, 1947, **42**, 26) gives results averaging $\simeq$45 per cent. of those obtained by the A.O.A.C. fermentation method. Chromatographic analysis after fermentation reveals complete removal of sucrose, glucose and (except occasionally for traces) fructose in the A.O.A.C. method. In the modified Serbia method, part of the unfermentable reducing matter (moving slowly on the chromatogram) passes the ion-exchangers, and is only partly removed by the Pb acetate treatment; these fractions probably include caramels, humic acid type of condensation products of sugar fragmentation products and unidentified matter. Allulose is found in very crude molasses. Variations in the amounts of acetoin formed by different yeasts would be a factor of uncertainty in the fermentation method. P. S. ARUP

1660. **A comparison of different methods for determining the water content of honey.** E. Abramson (*Mitt. Lebensm. Hyg., Bern*, 1953, **44** [6], 468–471).—The water content of 49 samples of commercial Swedish honey was determined by the following methods: (*i*) by drying *in vacuo* for 23 hr. at 70° C, (*ii*) by Karl Fischer titration, 0·1 to 0·2 g of honey being added dropwise to the dehydrated reagent mixture and the water being titrated by the dead-stop technique after 10 min. stirring, and (*iii*) by measurement of the refractive index and by conversion by means of the A.O.A.C. empirical table. Method (*ii*) gave the highest values and method (*iii*) the lowest. The experimental errors, as shown by variances calculated from replicates, were lowest in method (*iii*) and highest in method (*i*). A table is given which enables refractive index measurements to be converted to water-content values as determined by method (*i*) or by method (*ii*). E. HAYES

1661. **Rapid determination of chlorides in meat and fish products.** Z. Jedlinski (*Przem. Rol. Spoz.*, 1953, **7** [10], 365).—In order to find a reliable rapid procedure for the determination of chlorides in meat and fish products, the conventional analytical methods have been examined, including the titrimetric determination of NaCl (*i*) in the water extracts of meat and fish, (*ii*) in the ash obtained by incineration with Ca acetate and (*iii*) by W. J. Dyer's method with adsorption indicators. The A.O.A.C. method for the analysis of plants was adopted; it involves wet oxidation with HNO_3. The method was modified for the analysis of meat and fish products as follows. A 2 to 5-g sample, weighed to 0·01 g, is dissolved by heating in an Erlenmeyer flask with 10 ml of conc. HNO_3 and 0·1 g of $AgNO_3$ (15 to 20 min.). Eight ml of a 5 per cent. soln. of $KMnO_4$ are added and the product is heated on a water-bath for 30 min. After cooling to room temp., it is diluted with 40 ml of water; 2 ml of nitrobenzene and 2 ml of saturated ferric ammonium sulphate soln. are added. The soln. is titrated with 0·1 *N* potassium or ammonium rhodanide to a slight red-brown. The method is accurate to approx. $\pm$0·02 per cent. H. BURSTIN

1662. **Qualitative test for alkaline sulphite adulteration of meat. Application of the colour reaction of sulphites in presence of Bougault's hypophosphite reagent and sodium tungstate to a deproteinised extract of suspected meat.** L. Blanchard and J. Pantaleon (*Ann. Falsif.*, 1954, **47**, 32–33).—Detailed procedures are described for applying the specific colour test for sulphites (*cf.* Abstract 1502 above) to aq. macerations of meat after defecation by $Zn_2Fe(CN)_6$ by Carrez' method. The sensitivity of the test is 1 mg of Na_2SO_3 per sq. dm, when sulphite has been applied to the surface of meat-muscle, and $\simeq$0·025 per cent. (as Na_2SO_3) when it is mixed with the minced meat. W. J. BAKER

1663. **Colorimetric estimation of fat peroxides in meat.** L. Hartman, C. N. Hooker and H. E. Watt (*N.Z. J. Sci. Tech.*, 1954, **35** [4], 307–310).—A modification of the dichlorophenol-indophenol method of Loftus-Hills and Thiel (*J. Dairy Res.*, 1946, **14**, 340–353) with a *n*-propanol-xylene mixture as solvent is described. Phosphatides do not interfere and the fat sample can be extracted from the wet tissue. E. G. BRICKELL

1664. **Chemical evaluation of the freshness of raw pig fat.** J. Janicki, A. Rutkowski and B. Dierzynska (*Przem. Rol. Spoz.*, 1953, **7** [9], 306–308).—The deterioration of raw pig fat, before and after salting with 2·4 per cent. NaCl, as well as the deterioration of lard obtained by melting fresh pig fat on a water-bath, have been examined at 18° to 20° C over periods of 12 to 62 days. The deterioration of unsalted raw fat is the result of the hydrolysis of glycerides and the destruction of albuminoid substances; it is characterised by an increase in acid value from 0·42 to 8·31 after 12 days, and in peroxide value from 0·43 to 3·50 ml of 0·002 *N* $Na_2S_2O_3$ per g after 12 days. There is also an increase of NH_3 formation from 13·7 to 110 mg per

100 g and a reduction of pH from 6·9 to 6·0. Increasing change of colour with epihydrin aldehyde and neutral red are also observed. The corresponding increases for salted raw fat are: acid value from 0·44 to 0·97 after 12 days, to 1·82 after 26 days and to 4·67 after 62 days; peroxide value from 0·32 to 2·69 after 12 days, to 3·95 after 26 days and to 18·87 after 62 days; NH_3 from 14·1 to 38·1 mg per 100 g after 12 days, followed by a decrease to 27·4 mg per 100 g after 62 days. Organoleptic examination established unfitness for human consumption of raw fat and the corresponding lard after 7 days, and of salted raw fat after 26 days. Acid value was determined by Kreiss's method as modified by Kozin, peroxide value by the Wehler - Jacobs method, NH_3 colorimetrically with Nessler's reagent, and epihydrin by the Kreiss - Kozin test; the neutral red test was that used by Drozdov. The pH values were determined on the water extracts.

H. Burstin

1665. **Direct chromatographic determination of acetic, propionic and butyric acids in cheese.** W. J. Harper (*J. Dairy Sci.*, 1953, **36**, 808–816).—The direct chromatographic method proposed for separating and determining acetic, propionic and butyric acids in cheese does not require preliminary distillation or extraction; pyruvic acid is also separated. Recoveries of >90 per cent. were attained and titration results were reproducible within ±5 per cent. of the mean.

J. Sci. Food Agric. Abstr.

1666. **Acetaldehyde and related compounds in frozen green peas.** J. J. David and M. A. Joslyn (*Food Res.*, 1953, **18**, 390–398).—The chromatographic separation of 2:4-dinitrophenylhydrazones of carbonyl compounds occurring in fresh and frozen pea-seeds, their characterisation by absorption spectroscopy, and related observations are reported. The occurrence of acetaldehyde, pyruvic carboxylase, 3-hydroxybutanone and diacetyl are discussed. Ethanol was the only alcohol found in the steam-distillate of peas.

J. Sci. Food Agric. Abstr.

1667. **Determination of perchloroethylene in strawberries.** D. A. Mapes and S. A. Shrader (*J. Agric. Food Chem.*, 1954, **2** [4], 202–203).—A method is described for determining small quantities of residual perchloroethylene (C_2Cl_4) in strawberries that do not contain other chlorinated org. compounds: 100 g of fruit are shaken with 100 ml of ether in a sealed vessel, and, after leaving overnight, a 25-ml aliquot of the ether with 3 ml of ethylbenzene is evaporated to ≃ 3 ml, cooled and weighed (*A*). The solution is cooled in solid CO_2, 1 ml is weighed (*B*) into a cooled cup of a Parr bomb and ignited in the presence of 5 ml of 0·5 per cent. Na_2CO_3 solution under 500 lb pressure of O. The bomb contents are diluted to 50 ml with water and the Cl is determined nephelometrically on a 25-ml aliquot by adding 20 ml of Cl′-free alcohol and 5 ml of $AgNO_3$ soln. (1·75 g in 1 litre of water containing 12·5 ml of HNO_3), heating at 40° C for 15 min., cooling, and measuring the turbidity. The Cl′ content is obtained by reference to a standard graph. The amount of C_2Cl_4 (μg) in the fruit = (μg of C_2Cl_4 in final aliquot × 100 × 50 × *A* ÷ volume of final aliquot (ml) × 25 × *B* × wt. in g of original sample). Recovery of added C_2Cl_4 (0·023 — 0·18 mg) was 84 to 103 per cent.

S. C. Jolly

1668. **Detection of artificial aroma in natural extracts of coffee.** G. Séris (*Ann. Falsif.*, 1954, **47**, 26–29).—The presence of an aromatic adulterant in natural extracts of coffee is revealed by obtaining the u.v. absorption spectrum between 230 and 420 mμ of 2 ml of a *n*-hexane (vapour) extract of the volatile constituents in the coffee essence. A distinct max. at ≃315 mμ indicates addition of 0·1 to 1 per cent. of artificial aroma, the strong max. at 270 mμ corresponding to the natural aromatic volatiles. The sensitivity of the method is approx. 1 in 1000.

W. J. Baker

1669. **Colorimetric determination of trace metals in beer and in brewing materials. VII. Determination of nickel.** J. Andrews and G. A. F. Harrison (*J. Inst. Brewing*, 1954, **60** [2], 133–135).—The beer (100 ml), barley or malt (20 g), or hops or dried yeast (10 g) is oxidised by means of H_2SO_4 plus HNO_3 as previously described (*cf. Brit. Abstr. C*, 1952, 115). For beer, the total diluted oxidation mixture (or an aliquot representing 5 g of barley or malt, or 2 g of hops or dried yeast) is (with washings) mixed in a 150-ml separating funnel with 1 ml of aq. 10 per cent. w/v $K_2S_2O_5$, 10 ml of aq. 30 per cent. Rochelle salt and 10 ml of 3 *M* Na acetate, and the pH is brought (by test paper) to 8·0. After adding 5 ml of $CHCl_3$ and 2 ml of 1 per cent. ethanolic α-furildioxime, the total coloured matter is extracted with the 5 ml and subsequent smaller amounts of $CHCl_3$. The $CHCl_3$ extracts, made up to 20 ml, are freed from coloured Cu compounds by shaking with 50 ml of 0·005 *N* H_2SO_4, dried with anhydrous Na_2SO_4, and measured colorimetrically (Chance blue glass OB 10 or Ilford filter No. 621) in comparison with results obtained with a standard soln. containing 10 μg of Ni per ml. The range of the method is 0 to 50 μg of Ni.

P. S. Arup

1670. **Evaluation of kieselguhr as a beer filter-aid.** H. F. P. Webber and L. Taylor (*J. Inst. Brewing*, 1953, **59**, 392–397).—For the grading of kieselguhr a proposed scheme of analysis comprises macroscopical and microscopical examination and determinations of moisture content, loss on ignition at 600° C, water-solubles, pH in water, water-soluble Fe, As_2O_3 and Pb, 24-hr. sedimentation, effect on pH, colour and taste of beer, and rate of filtration. Typical data for good-quality Californian kieselguhr and for unsuitable brewery grades from Europe, Africa and Australia are appended.

J. Sci. Food Agric. Abstr.

1671. **Determination of nitrogen [in brewing materials].** E. Schild and L. Then (*Brauwissenschaft*, 1954, [2], 21–24).—Acetanilide, presumed to be suitable as a test substance for the Kjeldahl process, contained 1·11 per cent. of water; allowing for this, Weininger's rapid method (digestion for 20 min. with Na_2SO_4, $CuSO_4$ and Se catalyst) measures only 92 per cent. of the total N in acetanilide in presence of 1 g of sugar or starch. The values obtained are less than those obtained with acetanilide alone, even on digesting for 2 hr. with the addition of $HgSO_4$. The N contained in barley is more easily liberated than that in acetanilide, especially when the latter is mixed with sugar; for barley, the Weininger method with a digestion time extended to 1 hr. is sufficiently accurate in practice.

P. S. Arup

1672. **Further observations on the δ-resin of hops, and a method for its estimation.** J. W. Abson, M. S. E. Saleh and T. K. Walker (*J. Inst. Brewing*,

1954, **60** [1], 42–46).—The pptn. of α-soft resin of hops by Pb acetate is partly inhibited by the presence of the δ-resin, but proceeds satisfactorily after the removal of the bulk of the latter, by extracting the ether extract (250 ml, representing 25 g of hops) successively with 100 ml (for 5 min.), 50, 50 and 50 ml (each for 4 min.) of water. The combined aq. extract is extracted with light petroleum, b.p. $>40°$ C, to remove ether, and treated with charcoal; the δ-resin is then determined in 100 ml by rapid evaporation at 100° C under reduced pressure and weighing the residue after drying at 100° C. In order to allow for the incompleteness of the extraction (with water) of the δ-resin, the wt. found is multiplied by 1·282 when analysing hops not more than 3 months old, which contain 0·6 to 0·7 per cent. of δ-resin, or by 1·370 for hops 3 to 18 months old, which contain more (max. 4 per cent.) δ-resin. These factors are based on a few observations and may not be accurate for a larger number. Increases in δ-resin contents of hops are more rapid at warehouse temp. than in cold storage, and bear no relation to changes in soft-resin contents. P. S. ARUP

1673. **Titrimetric semimicro-determination of reducing sugars in wines and liquors.** A. J. Llacer (*Mikrochim. Acta*, 1954, [2], 193–200).—A semimicro modification of the Fehling - Causse - Bonnams method for the titrimetric determination of reducing sugars in wines and liquors is described. $K_4Fe(CN)_6$ is used to keep the Cu_2O in solution, and methylene blue is used as an internal indicator. The alkaline Cu soln. is composed of two solutions, one containing 12 g of $CuSO_4.5H_2O$ per litre and the other 65 g of Rochelle salt, 40 g of NaOH and 18 g of $K_4Fe(CN)_6$ per litre of H_2O; equal vol. of the two soln. are mixed before use, A fresh mixture should be made every day as it undergoes a slow change even if protected from air and light. It is standardised against a standard invert sugar soln. (5 g per litre, made from sucrose by treatment with conc. HCl and neutralising; phenol is added as a preservative). The burette is a modified Hybbinette and Benedetti-Pichler instrument. The liquid to be titrated is clarified with Pb acetate and decolorised if necessary with charcoal. A factor is applied to the final result to compensate for adsorption of reducing sugar by the charcoal (2 per cent. per 50 mg of adsorbent). The relative standard deviation is ±0·4 per cent. A. J. MEE

1674. **Determination of pH and buffering power of liquids such as wine by measuring the titration acidity and the alkalinity of the ash.** N. Roussopoulos (*Compt. Rend.*, 1953, **237**, 749–750).—The pH, titration acidity, and alkalinity of the ash of liquids, *e.g.*, wine, are expressed by the formula—

$$pH = pK - \log \{[\text{titration acidity (in milli-equiv.)}]/[\text{alkalinity of the ash (in milli-equiv.)}]\};$$

its buffering power can be calculated, independently of the dissociation constants of the acids present, by means of the formula—

$$dB/d(pH) = 2{\cdot}303\ (\text{titration acidity} \times \text{alkalinity of ash})/(\text{titration acidity} + \text{alkalinity of ash}),$$

where K is the dissociation constant of a single weak monobasic acid ($pK = -\log K$) and dB is the quantity of a strong base that, when added to the medium, brings the pH to d(pH).

J. SCI. FOOD AGRIC. ABSTR.

1675. **Determination of lactic acid in wines.** J. F. Casas Lucas (*Rev. Cienc. Apl.*, 1953, **7** [6], 526–529).—Polyhydroxy acids and polyphenols are removed by pptn. with basic Pb acetate. Volatile org. compounds (ethanol and acetaldehyde) are removed by distillation with aq. H_2SO_4. The sample is then oxidised with $Ce(SO_4)_2$ at the b.p., the acetaldehyde evolved is collected in a soln. containing $NaHSO_3$ and a phosphate buffer of pH 7, and estimated by neutralising the excess of bisulphite with 0·02 *N* I soln., adding solid $NaHCO_3$ and titrating the liberated bisulphite with 0·02 *N* I soln. Glycerol or butylene glycol do not interfere. Large amounts of sugar introduce a small error.

L. A. O'NEILL

1676. **Estimation of malic acid in wine by paper chromatography.** P. Ribéreau-Gayon (*Ann. Falsif.*, 1954, **47**, 3–9).—In a rapid, ascending paper-chromatographic method of estimating ≤ 2 μg of malic acid (or the completion of malolactic fermentation) in wine, without any preliminary treatment of the latter, a solvent mixture of *n*-propanol, eucalyptol and formic acid (5:5:2 by vol.) in equilibrium with water vapour is used. The spots (yellow on blue background) are revealed by spraying with an ethanolic soln. of bromophenol blue. The centre spot of each of the five chromatograms corresponding, respectively, to 2, 4, 6, 8 and 10 ml of wine is that of malic acid, and its intensity can be compared with that of the standard spots (equiv. to 1, 2 and 3 μg of malic acid) in order to calculate the malic acid content of the wine. The wine sample used for the spots ($\simeq 1$ ml) should contain <2 g of malic acid per litre, and it is preferable to prepare an approx. chromatogram first and then a more accurate one. Expose a paper strip (48 cm by 17 cm), accommodating five spots for each of four different wines plus the three standard spots, to the mixture of solvent and water vapour in a closed air-tight glass vessel and, after the solvent has ascended to $\simeq 15$ cm, remove the paper cylinder, dry the paper until free from formic acid and then develop the spots. W. J. BAKER

1677. **Detection and estimation of ethylenediaminetetra-acetic acid in wines.** G. Séris (*Ann. Falsif.*, 1954, **47**, 29–30).—To reveal the unauthorised presence of ethylenediaminetetra-acetic acid in wines, take 10 ml of filtered cold-decolorised wine, add 5 drops of acetic acid and 2 drops of 2 per cent. aq. $Co(NO_3)_2$, heat for 15 min., cool to 40° C, add 5 ml of 30 per cent. H_2O_2, heat carefully for a short time and then cool to room temp. The gradual appearance of a rose-violet colour, stable for several hr., indicates presence of the sequestrant, the amount of which may be estimated colorimetrically (max. absorption at $\simeq 540$ mμ). The procedure is sensitive qual. to 10 mg and quant. to 20 mg per litre. W. J. BAKER

1678. **New methods for the determination of glycerol in wine and similar products.** O. Reichard and H. Gspahn (*Z. anal. Chem.*, 1954, **141** [4], 252–272).—Published methods are placed in four groups according to their basic principle. One of these, based on the conversion of glycerol to acrolein is chosen for investigation as it promises greatest specificity. The estimation of acrolein by formation of benzanthrone and nitrophenylhydrazones is investigated but found unsatisfactory. By a modified Skraup synthesis, glycerol is converted to quinoline, which is determined gravimetrically as a mercuric iodide complex (empirical factor 0·11). Polyhydric alcohols, *e.g.*, butylene glycol, sorbitol, mannitol and other fermentation by-products do

not interfere, but sucrose in quantities greater than 20 g per litre must be removed by the barium hydroxide method.

Wines containing less than 20 *g of sucrose per litre*—Boil wine (10 ml) gently under reflux for 2 hours with aniline sulphate (2·0 g), Na *m*-nitrobenzenesulphonate (0·5 g) and conc. H_2SO_4 (11·0 ml), cool, dilute the mixture with 15 ml of water, make alkaline with 50 per cent. KOH soln. (40 ml) and steam distil, collecting 100 ml of distillate in 10 per cent. HCl (7 ml). Add 15 ml of quinoline reagent (100 g of KI in 200 ml of H_2O mixed with a soln. of 40·8 g of $HgCl_2$ in 500 ml of H_2O has 5 per cent. $HgCl_2$ added dropwise until a reddish opalescence forms when the soln. is diluted to 1 litre) and 5 per cent. $HgCl_2$ dropwise until the orange ppt. formed just disappears. Excess must be avoided. Filter precipitate, dry at 105° C and weigh.

(Wt. of ppt.) × 0·11 = wt. of glycerol.

Wines containing more than 20 *g of sucrose per litre*—To 30 ml of methanol in a 100-ml measuring flask, add 10 g of finely powdered $Ba(OH)_2$, shake, add slowly 40 ml of wine, set aside and make up to 100 ml with ethanol. After 1 hr. filter the mixture, evaporate alcohol from exactly 50 ml of filtrate and make up to 50 ml with water. Ten ml of this soln (≡4·0 ml of wine) is taken for determination as above.

Results on many varieties and types of wine are compared with results obtained by the periodate and calcium hydroxide method. P. S. Stross

1679. **Estimation of acetaldehyde in spirits.** P. Jaulmes and J. C. Dieuzede (*Ann. Falsif.*, 1954, **47**, 9–14).—A simple standard method of estimating total and free acetaldehyde in spirits is described. Add 10 ml of an acid soln. (pH 2) of $K_2S_2O_5$ to 50 ml of sample (adjusted to 50° alcohol) plus 300 ml of air-free water in a 500-ml flask with ground-glass stopper and, after shaking and sealing, leave the flask for ≃15 min. to complete the hydrolysis of the acetal. Add saturated aq. $Na_3PO_4.12H_2O$ (10 ml) and leave the liq. for ≃15 min. so as to effect combination between acetaldehyde and SO_2 at pH 3 to 4. Remove the excess of SO_2 by adding 10 ml of dil. HCl (1 + 4) and enough 0·1 *N* I soln. to give a blue coloration with starch. Titrate the soln. with 0·1 *N* I in presence of aq. $NaBO_2$ at pH 9·5. The *total* acetaldehyde in the sample = 2·24 *x* mg (2·28 *x* for rectified spirits), where *x* = ml of 0·1 *N* I required for titration of the combined SO_2. To determine the *free* acetaldehyde, mix the soln. of $K_2S_2O_5$ and Na_3PO_4 with ≃150 ml of water and add immediately the 50-ml sample of spirits, afterwards titrating as described above (free acetaldehyde = 2·2*x* mg). The prep. of the soln. and the precautions (*e.g.* control of pH) to be observed during the operations are described in detail. The accuracy of the results is about 1 per cent. and the lower limit of estimation is ≮0·5 mg of acetaldehyde per hectolitre of spirits. A slight modification is necessary for redistilled spirits. W. J. Baker

1680. **Colorimetric determination of chlorophyll in foodstuffs.** B. Wieclawek (*Przem. Rol. Spoz.*, 1953, **7** [9], 335–336).—This is a preliminary report on work carried out to establish standard testing methods for the determination of artificial dyestuffs in food. Pure K chlorophyllin was extracted from nettle, clover, lupine, peppermint and other plants, giving 0·13 to 0·37 per cent. of chlorophyll, calculated on dry substance; 0·7 to 1·0 per cent. was obtained from vegetable fats. Standard solutions were prepared of K chlorophyllin, Na chlorophyllin and K chlorophyllin - copper complex. These standard solutions served to establish the max. absorption at 650 mμ. The chlorophyll contents of various plants, artificially coloured fruit juices and fats were established by comparison with the standard solutions on a Coleman spectrophotometer. The extinction for filtered juice from greengage compote was 0·042, and from the curve established for a 0·5 per cent. w/v potassium chlorophyllin soln. without copper, a value of 0·800 was found with the 650-mμ filter. From this the K chlorophyllin content was 12·5 mg per 100 ml. The concn. of crude chlorophyll in unrefined rape-seed oil was found to be 41 to 42 mg per 100 ml, which is equivalent to 27·5 mg per 100 ml of pure chlorophyll. The method is applicable to the determination of traces of chlorophyll in foodstuffs and to the characterisation of natural chlorophyll in plants.

H. Burstin

1681. **Use of chromatography for detection and identification of dyes used for colouring foods.** G. Panopoulos and J. Mégaldoikonomos (*Chim. Anal.*, 1954, **36** [3], 68–69).—Amaranth (red No. 2), Ponceau Brilliant 4R and Ponceau 3R Extra can be identified, separately or when mixed, in red caviare by the two-dimensional strip-paper chromatographic procedure described. The solvents used are 1 per cent. aq. NH_3 saturated with *iso*pentanol and a mixture of *n*-butanol (200 ml), ethanol (40 ml), water (88 ml) and conc. aq. NH_3, sp. gr. 0·88 (2 ml); separation of the three dyes is complete in ≃ 7 hr. The prep. of the uncontaminated dye from the sample of caviare by successive extraction with ethanol and ether, followed by a no. of fixations on and solutions from pure fat-free wool to obtain finally a conc. soln. in 10 per cent. aq. NH_3 is described fully. By comparing the strip chromatograms obtained (under exactly similar conditions) with one drop of the dye extract and of each of the three dyes it is possible to determine whether one or more dyes is present in the extract and to identify the dyes, the R_F values for the ammonia - *iso*pentanol solvent being sufficiently different to ensure separate spots on the paper strip.

W. J. Baker

1682. **Colorimetric determination of propenylguaethol in imitation vanilla.** R. M. Roberts (*J. Ass. Off. Agric. Chem.*, 1953, **36** [4], 1119–1123).—Small quantities (2 to 10 mg) of propenylguaethol, coumarin and ethylvanillin are quant. steam-distilled from an acidified soln., the distillate being collected in a flask containing *iso*propanol; an aliquot is coupled with *p*-nitrobenzenediazonium chloride and the absorption of the resulting 2-ethoxy-4-*p*-nitrophenylazo-5-propenylphenol is determined at 530 mμ. Recoveries of 99·3 to 101·8 per cent. are reported. A. A. Eldridge

1683. **Separation and identification of coumarin and four other vanilla-like flavouring substances by paper chromatography.** L. C. Mitchell (*J. Ass. Off. Agric. Chem.*, 1953, **36** [4], 1123–1127).—A method is proposed for the separation by paper chromatography and identification of vanillin, ethylvanillin, coumarin, piperonal and Vanitrope (propenylguaethol; 2-ethoxy-5-propenylphenol, requiring mixed octanes as mobile solvent), and formamide dissolved in a mixture of ethanol and ether as stationary solvent. Aq. KOH (not applicable to

piperonal) and hydrazine sulphate in dil. HCl (not applicable to coumarin and Vanitrope) are used as chromogenic agents. A. A. ELDRIDGE

1684. **Contribution to the determination of peroxide numbers.** H. Hadorn and R. Jungkunz (*Mitt. Lebensm. Hyg., Bern.*, 1953, **44** [6], 495–500).—A modification of the Lea method is proposed in which the quantity of KI is reduced and the whole operation is carried out in one vessel. Place 1 g of the oil or fat in a 100-ml ground-glass flat-bottomed flask, add 0·1 g of finely powdered KI and then 20 ml of acetic acid - chloroform (1 + 1 by vol.). Fit the flask with an upright tube, 40 cm long and 10 mm bore, and, by means of a 5-mm tube reaching to the lower end of the ground-glass joint, pass a stream of CO_2 into the flask for 2 min. Remove the inlet tube and heat the flask with frequent shaking on a bath of boiling water until the mixture boils vigorously. Cool rapidly, remove the upright tube and add 40 ml of 0·2 per cent. KI soln. and 0·5 ml of 1 per cent. starch soln. Titrate the liberated iodine with 0·01 *N* $Na_2S_2O_3$ avoiding direct sunlight. Carry out a blank titration without the oil; with pure reagents this should be zero. Peroxide number = (ml of 0·01 *N* iodine) × 5. The results on various oils show good agreement with those found by the standard method. If the air in the flask is not displaced by CO_2, the results are too high. Good fresh oils usually have peroxide numbers <3; an exception is olive oil, which normally gives values of from 8 to 12. E. HAYES

1685. **Detection and approximate determination of cruciferous [-plant] oils in edible oils by the lead salt procedure.** H. Hadorn and R. Jungkunz (*Mitt. Lebensm. Hyg., Bern*, 1953, **44** [6], 453–466).—The various methods proposed by Grossfeld were examined and found to be unsatisfactory; high erucic acid numbers, *i.e.*, the amount of 0·1 *N* iodine (ml) required for the titration of the Pb salt separated from 500 mg of oil, were obtained with some non-cruciferous oils. The following modified method gives satisfactory results. Gently reflux 500 mg of oil with 5 ml of 0·5 *N* ethanolic KOH in a 50-ml flat-bottomed flask for 15 min. To the warm soln. add 20 ml of Pb acetate soln. (12·5 g of Pb acetate crystals plus 2 ml of 96 per cent. acetic acid made up to a litre with 80 per cent. v/v aq. ethanol), 1 ml of acetic acid and 3 ml of water. Heat under reflux until clear; cork and set aside overnight at 20° C. Filter on a Buchner funnel through a suitable 4-cm filter previously moistened with 70 per cent. aq. ethanol. Transfer the residue to a 300-ml Erlenmeyer flask and dissolve it in 20 ml of ethanol - acetic acid (equal vol. of 96 per cent. ethanol and 96 per cent. acetic acid), heating to boiling. Draw the filter to the side of the flask with a wire hook, wash it with warm ethanol and remove it. Into the cooled fatty acid soln., pipette 20 ml of 0·2 *N* ethanolic iodine (25·4 g of iodine and 96 per cent. v/v ethanol to a litre), mix and immediately add 150 ml of water. Set the soln. aside in the dark for 3 to 5 min. and then titrate with 0·1 *N* $Na_2S_2O_3$, using 0·2 per cent. starch soln. as indicator; carry out a blank titration with 20 ml of ethanol - acetic acid and 20 ml of iodine soln. By this method edible oils give erucic acid numbers of <1·0; for mustard oils the values are 9 to 10 and for rape oils 10 to 13. The method can be used to detect the presence of rape oil in edible oils when it is present in amounts greater than 20 per cent. E. HAYES

1686. **Chemistry of the Baudouin reaction [for sesame oil].** L. B. Mathur, R. Sahai and R. N. Mathur (*J. Oil Technol. Ass. India*, 1952, **8**, 41–47).—The chemistry of the Baudouin colour test for the determination of sesame oil is discussed and experimental modifications are suggested. The colour intensity produced is proportional to the sesamol content of the oil. The oil (20 ml) is diluted with liquid paraffin (80 ml). The mixture (5 ml) is heated to 60° C and conc. HCl (5 ml) and a 2 per cent. alcoholic soln of furfural (0·5 ml) are added. The mixture is shaken vigorously, allowed to separate and after filtration the colour of the lower layer is recorded. Under these conditions max. colour intensity is attained. N. M. WALLER

1687. **Relative viscosity as a purity test for mustard oil.** S. N. Mitra and S. C. Roy (*Curr. Sci.*, 1954, **23** [2], 50–51).—Relative viscosities at 40° C of mustard oil (60·2 to 63) and 8 other vegetable oils are given. Adulteration of the mustard oil results in a decrease of the relative viscosity, and 59·0 is suggested as the lowest permissible figure for the genuine oil. D. BAILEY

1688. **Iodimetric determination of ascorbic acid in dried rose hips.** K. Backe-Hansen and A. Nordal (*Dansk Tidsskr. Farm.*, 1954, **28** [3], 53–63).—The methods of the Swedish and Danish Pharmacopoeias are examined. The following modified method is proposed as an addendum to the Norwegian Pharmacopoeia (1939): a mixture of the powdered sample (1 g) with boiling water (100 ml) and 4 *N* acetic acid (1 ml) is boiled for 10 min. and rapidly cooled, and the extract, mixed with 2 *N* H_2SO_4 (10 ml), is immediately slowly titrated with 0·1 *N* I soln. (1 ml ≡ 0·0088 g of ascorbic acid) in presence of 5 ml of 1 per cent. starch mucilage until the blue colour persists for ⩽30 min. The provision of an atmosphere of CO_2 over the liquid during extraction and titration is unnecessary. P. S. ARUP

1689. **Nitrosation method of determining D-γ-tocopherol.** B. H. Polister (*Anal. Chem.*, 1954, **26** [2], 407–408).—D-γ-Tocopherol is determined by nitrosation in 1:2-dimethoxyethane, extraction of the colour by *iso*octane and determination of the absorption at 305 and 415 mμ. A soln. (5 ml) of tocopherol in dimethoxyethane is treated with 0·2 ml of acetic acid and 3 ml of 2 per cent. aq. $NaNO_2$. The mixture is swirled for 5 sec. and set aside for 60 sec., when 2 ml of KOH soln (20 g of KOH in 100 ml of H_2O), 10 ml of H_2O, a little anhyd. Na_2SO_4 and 10 ml of *iso*octane are added. After shaking for 30 sec. and allowing to settle, the absorption of the *iso*octane layer is determined on a recording spectrophotometer at 450 to 350 mμ and 340 to 280 mμ and the reading is compared with that of a blank. D. BAILEY

1690. **The separation and determination of pteroylglutamic acid and related compounds.** S. F. Zakrzewski and C. A. Nichol (*J. Biol. Chem.*, 1953, **205** [1], 361–368).—Pteroylglutamic acid and Aminopterin (the 4-amino analogue) can be separated by paper chromatography. None of the org. solvents used for chromatography of pteridines is satisfactory, but good resolution is obtained with 0·1 *M* phosphate buffer from pH 6·2 to 7·7, or with 0·1 *M* acetate buffer at pH 6·3. A fluorimetric assay of Aminopterin and Amethopterin (4-amino-10-methylpteroylglutamic acid) involves (*i*) alkaline

hydrolysis, which replaces the 4-amino by the 4-hydroxy group, and (*ii*) oxidation with $KMnO_4$ to a product, presumably pteroyl-6-carboxylic acid, which has the same intensity of fluorescence as the product derived similarly from pteroylglutamic acid. Analysis of a sample of Aminopterin showed 24 and 22 per cent. of pteroylglutamic acid by the fluorimetric and microbiological methods, respectively.
J. N. ASHLEY

1691. **Vitamin assay. Rapid spectrophotometric determination of vitamin B_{12} in microbial material.** R. A. Fisher (*J. Agric. Food Chem.*, 1953, **1**, 951–953).—Dry material is extracted with hot benzyl alcohol containing CN' and H_2O, pH 7 to 8; with moist material, *n*-propanol is used. The vitamin is then determined by differential spectrophotometric measurement. Five tables of comparative data are given. J. SCI. FOOD AGRIC. ABSTR.

See also Abstract 1502.

Sanitation

1692. **Some new physico-chemical methods for the analysis of waters.** K. A. Murray (*S. Afr. Ind. Chem.*, 1952, **6**, 270–272).—This preliminary note describes the development of physico-chemical methods for water analysis. A potentiometric method for the determination of nitrite in soln. is based on the determination of the iodine - iodide potential against the S.C.E. To 10 ml of 0·01 *M* KI soln. are added 5 ml of the nitrite soln. The mixture is maintained at 25° C and pure N is bubbled through to remove all O_2 which interferes; 5 ml of *N* H_2SO_4 are added and the e.m.f. is measured after 10 min. Nitrite at concn. as low as 0·1 p.p.m. of nitrogen can be determined. A method for the determination of dissolved O was developed from the above method. A sample of water containing dissolved O is added to 0·02 *M* KI soln. immediately after the addition of the H_2SO_4. The reaction is not rapid, but readings taken after 10 min. give a value of 60 per cent. of the total O concn. with reproducible accuracy of ± 5 per cent. A manometric method for the determination of sulphide is based on its catalytic effect on the reaction between azide and I. The volume of N evolved is proportional to the sulphide present and independent of the I. The reaction should be carried out at pH 6. For concn. of sulphide in the range 0·5 to 0·01 p.p.m., the standard deviation was found to be about 10 per cent. of the sulphide concn. Sulphide can also be determined amperometrically by the same reaction. Electrically generated I is used to titrate the azide ion under the catalytic influence of sulphide. The end-point, which is reached when all the sulphide has been destroyed by I, is detected amperometrically. Sulphide can be determined in concn. between 0·01 p.p.m. and 0·08 p.p.m. With platinum electrodes and a 70-ml sample, 5 ml of *N* KI, 5 ml of *N* sodium azide and 1 ml of 0·17 *N* HCl (pH 6) are added. A volumetric method for determining K by dilituric acid is being devised. A manometric method is being devised for distinguishing nitrified sewage-works effluents from poorly oxidised effluents or from dil. untreated sewage and consists in measuring with a Barcroft respirometer the uptake of O of a 5-ml sample to which have been added 0·5 ml of a double strength McConkey broth and 1·0 ml of 8 per cent. $NaNO_2$ soln. Applications of these analytical methods are indicated.
WATER POLLUTION ABSTR.

1693. **Complexometric determination of hardness of water.** A. Smola and G. Hofbauer (*Mitt. chem. Forsch.Inst. Wirt. Öst.*, 1954, **8** [1], 12, 14–17).—Ca plus Mg can be determined in water, after adjustment to pH 12 and addition of Eriochrome black T (specific for Mg), by titration with sodium ethylenediaminetetra-acetate (**I**). Ca only can be determined similarly at pH 10 with murexide (specific for Ca). It is normally recommended that the titration should be done above 40° C, but $CaCO_3$ is liable to be pptd. Preliminary acidification and removal of CO_2 by boiling prevents this, but increases the time required. It is shown that satisfactory results can be obtained by slow titration at room temp., or by addition of excess of **I** at room temp., and back-titration with the water at 40° C.
A. B. DENSHAM

1694. **Determination of sodium and potassium in natural waters.** M. V. Tovbin and F. G. Dyatlovitskaya (*Ukr. Chem. J.*, 1952, **18** [6], 657–660).—The $Na^{\cdot}$ plus $K^{\cdot}$ content of the water is exchanged on a cationite for $H^{\cdot}$, which are titrated in the effluent from the column. The results are $\simeq$3·5 per cent. low owing to incomplete exchange. R. C. MURRAY

1695. **Use of organolites (ion-exchange resins) in water analysis.** M. V. Tovbin and F. G. Dyatlovitskaya (*Ukr. Chem. J.*, 1952, **18** [6], 647–656).—Optimum conditions are worked out for the quant. determination of $NH_4^{\cdot}$ and $Fe^{\cdot\cdot\cdot}$ plus $Fe^{\cdot\cdot}$ in natural waters, by fixation on cationites, release by washing with HCl, and chemical determination in the wash water. Only total Fe can be determined, because $Fe^{\cdot\cdot\cdot}$ is partly reduced by normal cationites.
R. C. MURRAY

1696. **A comparison of the oxygen consumed from permanganate and B.O.D. tests in the study of textile wastes.** Rhode Island Section A.A.T.C.C. Sub-committee on Stream Pollution (*Amer. Dyest. Rep.*, 1954, **43** [5], P 130–P 131).—In a series of tests to compare the biological oxygen demand (B.O.D.) test and the oxygen consumed (OC) test for measuring the polluting effects on streams of wastes causing oxygen depletion, it is concluded that both tests have their place in the study of textile wastes. The OC test gives results dependent on the conditions used. Reaction time, temp., and concn. of reactants should always be noted. The B.O.D. test requires more time and equipment, but gives more satisfactory and significant results. The OC test is useful for specific purposes where time is an important factor (*e.g.*, trickling filter failure). Data given indicate that some textile wastes have a greater O absorbing effect per gall. on streams than domestic sewage. E. S. LANE

See also Abstracts 1494, 1555.

Agriculture and Plant Biochemistry

1697. **Rapid micro-volumetric determination of potassium in plants using sodium tetraphenylboron as precipitant.** A. M. Amin (*Chemist Analyst*, 1954, **43** [1], 4–6).—The ppt. formed when $NaB(C_6H_5)_4$ is added to a weakly acidic solution of $K^{\cdot}$ is used to estimate the K in tobacco. Heat the sample at 400° ± 10° C for 30 min. and treat the charred residue with 0·05 *N* HCl and filter. Reignite the ppt. and filter-paper at 600° C and extract as before. Dilute the combined extracts to 100 ml. Ppt. the K in 1 to 2 ml of the extract with a 3 per cent. aq. $NaB(C_6H_5)_4$. Cool, filter and wash. Dissolve the ppt. in acetone and, after evaporating and igniting,

titrate to methyl red with standard alkali or dissolve in a little sat. aq. $HgCl_2$ soln. and add a known vol. of 0·01 *N* NaOH, adding methyl red and heating to boiling (if indicator turns red add more 0·01 *N* NaOH). Boil, add 20 per cent. aq. KI until the ppt. redissolves, and titrate with 0·01 *N* HCl. Boil to expel CO_2 and adjust to the end-point with a further quantity of 0·01 *N* NaOH. Results for both methods are given, and compare well with the cobaltinitrite method. G. B. THACKRAY

1698. **The determination of lignin in plant tissues: use of a continuous-extraction method to remove interfering materials.** D. MacDougall (*J. Sci. Food Agric.*, 1954, **5** [2], 103–107).—A simplified pretreatment for lignin by continuous acid extraction is described, which is readily adaptable to routine analytical determinations. A Soxhlet extractor is used with a 1-litre boiling flask, and the strength and vol. of the HCl are adjusted to give a concn. of 1 per cent. w/w passing through the material in alundum or paper thimbles. A 6-hr. extraction by this method gives a lignin value similar to that obtained by the 3-hr. standard refluxing method, but the lignin so isolated is lower in N and methoxyl content. A 9-hr. extraction removes almost all acid-sol. material. N. M. WALLER

1699. **A comparative study of the methods for determination of lignin in esparto grass.** A. Soler and A. Carrasco (*An. Soc. Esp. Fis. Quim., B*, 1954, **50** [1], 99–106).—The lignin content as estimated by the Norman and Mitchell method (involving pre-hydrolysis) was lower than that given by the Ritter method (no pre-hydrolysis). An examination of the reducing power of the hydrolysis liquids, and also of the apparent loss of methoxyl and the content of hydroxyl groups in the lignin separated by each process indicated that the second method gave too high a value. None of the known methods of estimation of lignin are considered to be accurate. M. TADMAN

1700. **Identification and colorimetric determination of the oxidases of the corn-root tip.** E. L. Ginter and F. G. Smith (*Iowa St. Coll. J. Sci.*, 1953, **28** [2], 177–188).—The colorimetric method for cytochrome oxidase of Smith and Stotz (*Brit. Abstr. C*, 1949, 346) has been adapted for the assay of plant material. It is shown to give rates proportional to enzyme concn. over a sufficiently wide range for assay purposes, but precautions are necessary to avoid interference. If peroxidase is present, catalase treatment of the leuco dye is necessary. The concn. of the leuco dye, the oxidised dye and cytochrome-*c* must be kept within certain limits as they influence enzyme activity. Experiments to identify and measure the activity of the oxidases of corn-root tips are described. N. M. WALLER

1701. **Use of sulphuric acid in the detection and estimation of steroidal sapogenins.** H. A. Walens, A. Turner, jun., and M. E. Wall (*Anal. Chem.*, 1954, **26** [2], 325–329).—A rapid method for the identification and estimation of steroidal sapogenins is described. The steroidal sapogenin (0·1 to 5·0 mg) is dissolved in 94 per cent. H_2SO_4(10 ml) and warmed at 40° C for 16 hr., and the u.v. absorption spectra, which are characteristic, are determined in the 220 to 400-mμ region. Constituents of binary mixtures can be spectrophotometrically determined from appropriate equations based on previously determined absorptivities. More complex mixtures can be determined after preliminary chromatographic separation. A qual. scheme is suggested for the identification of 13 common steroidal sapogenins from chromatographic behaviour, spectra of the H_2SO_4 chromogens and m.p. D. BAILEY

1702. **Polarographic nitrate determination in plant-physiological investigations.** D. Munsche (*Z. PflErnähr. Düng.*, 1953, **62**, 229–239).—Plant extracts are satisfactorily freed from interfering organic substances of high mol. wt. (especially polypeptides and peptones) before nitrate determination by the uranyl acetate method of Kolthoff, Harris and Matsuyama (*J. Amer. Chem. Soc.*, 1944, **66**, 1782), by passage (dropwise) through a column (20 cm by 1·5 cm bore) of the exchange resin Wofatit F. Results agree well with those obtained by the colorimetric xylenol method. Further applications of the method include soil analysis. J. SCI. FOOD AGRIC. ABSTR.

1703. **Determination of phosphoric acid in plant ash by the "Photo-Rex" method.** P. Laske (*Z. PflErnähr. Düng.*, 1953, **62**, 38–41).—A modification of Lederle's method (*cf. Brit. Abstr. C*, 1948, 214) embodies the use of 3 per cent. of citric acid (instead of 1 per cent.) in developing the colour, in order to stop interference from large amounts of SiO_2. J. SCI. FOOD AGRIC. ABSTR.

1704. **Synthesis of cellulose by *Acetobacter xylinum*. I. Micro-method for the determination of celluloses.** M. Schramm and Shlomo Hestrin (*Biochem. J.*, 1954, **56** [1], 163–166).—A method for purification and quant. recovery of small amounts of bacterial cellulose is described. A relatively rapid and specific micro-method for determination of cellulose is given; the error is $\pm$4 per cent., and the lower threshold value is 20 μg. It depends on solution of the purified polysaccharide by acetolytic degradation with acetic anhydride - acetic acid - H_2SO_4, sp. gr. 1·84 (1:1:0·05 by vol.), conversion of the product to reducing sugar by acid hydrolysis, and colorimetric determination of the sugar by means of a Cu reagent. J. N. ASHLEY

1705. **Trichloroacetic acid. Colorimetric method for quantitative determination in plant tissue.** T. W. Tibbitts and L. G. Holm (*J. Agric. Food Chem.*, 1953, **1**, 724–726).—The plant tissue is treated with 0·1 *N* acetic acid and filtered through asbestos. An aliquot of the filtrate is added to a tube containing pyridine and NaOH. The soln. is heated to a red-violet colour, which is measured colorimetrically. Quantities as low as 25 μg per g of fresh tissue can be determined in this way. J. SCI. FOOD AGRIC. ABSTR.

1706. **Determination of carotene in fodder and foods.** R. Bürke (*Z. PflErnähr. Düng.*, 1953, **62**, 193–210).—A review, including details of methods used by the authors. J. SCI. FOOD AGRIC. ABSTR.

1707. **Chromatographic method of determining raffinose in fodder molasses.** A. K. Kartashov and V. A. Serdyuk (*Sakhar. Prom.*, 1953, [11], 19–22).—The determinations of raffinose in beets and in molasses by de Whalley, Albon and Gross are summarised. Tests show that satisfactory separations can be made after demineralising the diluted molasses with ion-exchange resins and using water-saturated phenol as solvent. Results are best when

the molasses is diluted to contain 2 to 5 per cent. of dry solids and the paper is spotted with $<0{\cdot}01$ mg of soln. All filter-papers tested were satisfactory and constant R_F values were attained. The tests have so far been only qualitative. The best spraying reagent is a 0·3 to 0·6 per cent. soln. of 1-naphthol with 10 per cent. v/v of orthophosphoric acid added just before use. SUGAR IND. ABSTR.

1708. **Determination of free iron oxide in soil and clay.** A. Karim (*Pakistan J. Forestry*, 1953, **3**, 48–51).—The method of Jeffries (*Soil Sci.*, 1941, **52**, 451) is modified. Fe_2O_3 in the ground sample is reduced by Zn dust in a mixture of oxalic acid and potassium hydrogen oxalate (pH 3·5), and Fe is subsequently titrated with $K_2Cr_2O_7$; if >1 per cent. of chalk is present in the original soil, it must be removed by 0·1 *N* HCl.

J. SCI. FOOD AGRIC. ABSTR.

1709. **Conductimetric determination of exchange capacity of soils and their constituents.** H. Sandhoff *Z. PflErnähr. Düng.*, 1953, **62**, 1–19).—The air-dried sieved sample (0·25 to 3 g, corresponding to 25 to 2 per cent. of humus) is shaken for 1 hr. with 0·01 *N* NaOH (100 ml) and, if the humus exceeds 2 per cent., warmed to $>50°$ C for 30 min. After setting aside overnight, the suspension is acidified with dil. HCl (1 + 1) (20 ml), filtered and washed with dil. HCl (1 + 1) (50 ml) and then with water until all Cl′ has been removed. If alkali tratment is omitted, dialysis with water for 24 hr. will be necessary at this stage, whilst the conductimetric titration is apt to be retarded. The conductimetric titration vessel (described) is fitted with a glass electrode for simultaneous determination of pH. On titration with 0·2 *N* $Ba(OH)_2$ (0·1 to 0·2 ml additions at a time), the conductimetric titration curve, after an initial fall, remains nearly horizontal and then rises steeply. Evaluation of the flat portion of the curve, in ml of 0·2 *N* $Ba(OH)_2$, gives a measure of the *T* value. Data are given for various soils, with or without additions of lime or peat. Comparisons with results by the Jensen and the Mehlich - Schachtschabel method are given.

J. SCI. FOOD AGRIC. ABSTR.

1710. **The 1:1′-dianthrimide method for determination of boron in soils; further observations.** E. Gorfinkiel and A. G. Pollard (*J. Sci. Food Agric.*, 1954, **5** [3], 136–139).—Quantities of water-soluble boron of the order 0·3 p.p.m. (dry soil) are determined by a modification of the 1:1′-dianthrimide method. The coeff. of variation of results is 5·8 per cent. and the standard deviation is 0·178. A 20-g soil sample, air-dried and passing a 20-mesh sieve, is extracted by boiling for 5 min. with 40 ml of H_2O under a reflux condenser. To the cooled extract, 5 ml of saturated soln. of NaCl are added and the soln. is centrifuged for 15 min. at 5500 r.p.m. Twenty ml of the supernatant liquid are decanted, 10 ml of saturated $Ca(OH)_2$ soln. are added and the soln. is evaporated to dryness. The original method (*Brit. Abstr. C*, 1953, 277) is then used, 2 ml of the final test soln. being used and the reagent containing 600 mg instead of 400 mg of 1:1′-dianthrimide in 98 per cent. H_2SO_4. The sensitivity of the method is increased by use of an orange filter and is greater than that of the Waxoline purple method.

N. M. WALLER

1711. **Electrochemical methods for measurement of soil aggressivity.** T. Markovič (*Nafta*, 1953, [9], 299–303).—Two methods for measuring rates of corrosion of metals in soils by means of a platinum auxiliary electrode and the Fe (rough surface) - wet soil - Fe (smooth surface) system are discussed. Measurements with Pt show that there exists an active type of corrosion when the soil is completely covered with water. With the Fe / soil / Fe device, the effect of electrolytes on the increase of corrosion intensity was investigated, the time necessary for the compensation of the electrode potential giving a measure of the effect.

J. SCI. FOOD AGRIC. ABSTR.

1712. **Emulsion testing. Basis for tests of emulsifiable concentrates of agricultural chemicals.** R. W. Behrens and W. C. Griffin (*J. Agric. Food Chem.*, 1953, [1], 720–724).—Data are presented indicating the effects of mode of addition of ingredients, agitation, and other variables on the laboratory prep. of emulsions from emulsifiable concentrates. A test method is described. Standardisation is attained by using a commercial emulsion viewer and 50-ml Nessler tubes filled to a depth of 7⅞ in.; the test emulsion is examined by uniform transmitted light. J. SCI. FOOD AGRIC. ABSTR.

See also Abstracts 1528, 1546, 1602, 1658.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1713. **Apparatus for determining gases contained in metals.** Vereinigte Osterreichische Eisen-und Stahlwerk A.-G. (Brit. Pat. 706,459, Date Appl. 27.5.52, Austria 8.9.51).—After evacuation, *e.g.*, by means of a mercury pump, and heating in an electric oven, the gases developed from the specimen are adsorbed by an adsorbent (activated C or dehydrated zeolite) that is cooled, *e.g.*, in liquid air. The gases are released from the adsorbent by removal of the liquid air and conveyed by the mercury pump through a pneumatic trough into the collecting vessel. J. M. JACOBS

1714. **Ball and socket nitrometer with integral liquid-levelling device.** E. Stehr (*Mikrochim. Acta*, 1954, [2], 213–218).—An improved micro-nitrometer for the Dumas-micro method is described. It has a ball and socket valve to replace the conventional stopcock at the top of the graduated column and an integral liquid-levelling device, which obviates the need for a levelling bulb and rubber connection with the nitrometer. Contamination from stopcock grease and materials from the rubber tube is thus avoided. A. J. MEE

1715. **Apparatus for rapid micro-determination of nitrogen.** A. Budziszewski (*Roczn. Chem.*, 1954, **28** [1], 145–147).—A Kjeldahl flask has its neck bent at a right angle to the bulb (150 ml), and a spherical splash head is fitted to the end of the neck by means of a ground-glass joint. A cylindrical 10-ml tap funnel is sealed to the top of the splash head, and a horizontal air cooler tube is sealed to its side. The downwards bent end of the air cooler is connected with the receiver, a 100-ml round-bottomed flask, by means of a rubber stopper, and a side branch leads to a vacuum pump. The substance containing 0·2 to 0·6 mg of N is treated with H_2SO_4 in the Kjeldahl flask. Conc. NaOH is placed in the tap funnel and excess of standard H_2SO_4 soln. in the receiver. The apparatus is assembled, evacuated and, by careful turning of the tap, excess of NaOH is sucked into the flask. By

immersion of the spherical part of the flask in boiling water and the receiver in cold water, the NH_3 is quantitatively distilled over in 3 min. The apparatus is convenient for serial analyses; ten units assembled into one battery enable one person to carry out up to 60 distillations per hr.

H. BURSTIN

1716. **Determination of nitrogen in organic compounds. A simple electrolytic generator for oxygen and a convenient form of flowmeter.** F. R. Cropper, R. H. Reed and R. Rothwell (*Mikrochim. Acta*, 1954, [2], 223–225).—A simple method of obtaining a steady current of O for use in the Dumas micro-method of determining N is described. It involves electrolysis of *M* H_2SO_4 with platinum-wire electrodes. A flowmeter with an adjustable range is also described. It is inserted between a "Drikold" CO_2 generator and the oxygen generator, so that controlled rates of flow of CO_2 can be used.

A. J. MEE

1717. **An apparatus for delivering vapour of constant composition and pressure.** A. Charnley, G. L. Isles and J. S. Rowlinson (*J. Sci. Instrum.*, 1954, **31** [4], 145–146).—The liquid to be vaporised is forced via a capillary tube into a "flash" chamber heated to a temp. 50° higher than the boiling point of the liquid. The liquid immediately vaporises and is stored in a heated bulb. This device ensures that the composition of the vapour is constant even if the volatilities of the components are widely different.

G. SKIRROW

1718. **An easily constructed manometer.** A. J. Castro and A. E. Blood (*J. Chem. Educ.*, 1954, **31** [1], 23).—The construction of a simple inexpensive manometer for rough measurements in the range 1 to 110 mm of mercury is described. The design is conventional and the apparatus comprises an outer Pyrex-glass test tube, 150 mm × 18 mm diam., fitted with a side-arm and a rubber bung, through which passes a 6-mm diam. Pyrex-glass tube sealed at a level just below the bung and drawn out to a 0·5 mm aperture at the bottom end, which reaches almost to the base of the outer tube. The method of filling the inner tube with mercury is described.

G. HELMS

1719. **A piezo-electric recording manometer.** S. N. A. Margerson and H. Robinson (*Minist. Fuel Pwr. Safety Min. Res.*, 1954, *Res. Rep.* No. 82, 34 pp.).—A manometer for recording explosion pressures during the flameproof testing of flameproof housings (for use in coal mines and in industrial processes where the ignition of inflammable gases or vapours is a hazard) comprises a quartz piezo-electric gauge and an impedance converter, by means of which the output of the gauge is transferred to the amplifier of the recording oscillograph. The design of the components and the calibration of the gauge are discussed, together with the application of the device to pressure development under pressure-piling conditions and in oxygen breathing apparatus, to the bursting pressure of Perspex tubes, etc.

J. M. JACOBS

1720. [**Gas-bell**] **manostats.** Emil Greiner Co. (Brit Pat. 706,777, 19.2.51, U.S. Pat. 1.9.50).—Structural details are claimed of a manostat able to "memorise" a particular vacuum or pressure at which it has been operating and to which it returns the system after the plant has been shut down.

J. M. JACOBS

1721. **A phase-study apparatus for semi-micro use above atmospheric pressure.** W. L. Marshall, H. W. Wright and C. H. Secoy (*J. Chem. Educ.*, 1954, **31** [1], 34–36).—An apparatus is described whereby phase equilibria at pressures above atmospheric can be studied with relative ease and time economy on semi-micro amounts of soln. By comparison with apparatus needed for macro studies, investigations can be made at much higher pressure, tube explosions are non-existent at pressures up to at least 3200 lb per sq. in.; temp. as high as 600° C with H_2O as the supercritical fluid have been used without an explosion. Experimental data on the system Na_2SO_4 - H_2O from 250° to 355° C are given.

G. HELMS

1722. **A thermistor McLeod gauge for a pressure range 1 to 10^{-7} mm of mercury.** R. S. Bradley (*J. Sci. Instrum.*, 1954, **31** [4], 129–130).—A thermistor bead sealed into the tip of the closed capillary of a McLeod gauge permits pressures of non-condensible gases in the range 1 to 10^{-7} mm of mercury to be measured.

G. SKIRROW

1723. **Apparatus for measuring the density of liquids.** R. Pochan and A. Pochan (Brit. Pat. 707,118, 13.3.52, Fr. Pat. 17.3.51).—A transparent tube is adapted to contain the liquid and the hydrometer. A circular line marked on the tube serves as a reference mark for reading the hydrometer graduations. Electrical means are provided for bringing the liquid to a level at a fixed distance from the reference mark. The hydrometer ballast, in the form of lead shot, is agglomerated so that a vertical floating position is ensured, and, by providing lateral projections on the hull, rotation of the hydrometer is prevented. The stem is graduated in mm and is viewed through a lens. Accuracy to the fourth decimal place of density, when a thermostatic jacket is used is claimed.

J. W. MULLIN

1724. **A tap for corrosive vapours.** A. B. Osborn (*J. Sci. Instrum.*, 1954, **31** [4], 147).—The tap which shows negligible leakage under pressure differences of one third of an atmosphere is constructed from a precision bore glass barrel in which a polythene or polytetrafluoroethylene plunger slides. The plunger is made slightly oversize and makes ring contact with the barrel.

G. SKIRROW

1725. **A simply-made micro-burner.** S. M. Charlett (*Lab. Practice*, 1954, **3** [4], 162).—A small jet for a burner for micro-manipulation is constructed from a hypodermic needle cut off and ground to have a square end.

G. SKIRROW

1726. **A cheap hot stage.** C. C. Kiplinger (*J. Chem. Educ.*, 1954, **31** [1], 33).—The construction and operation of an electrically heated air-bath m.p. apparatus, based on the Walter heater unit (*Brit. Abstr. C*, 1953, 327), are described.

G. HELMS

1727. **An apparatus for the wet ashing of organic matter.** P. O. Bethge (*Anal. Chim. Acta*, 1954, **10** [4], 317–320).—The apparatus provides for the condensation of vapours evolved during wet oxidations; the condensate can either return to the reaction flask or be reserved for separate examination. The condenser exit is fitted with a trap so that gaseous reaction products can be washed with appropriate reagents. A general method of use with mixed $HClO_4$ and HNO_3 is described with particular reference to the determination of S in organic matter.

W. C. JOHNSON

1728. **A gassing chamber for use with the Haldane method of haemoglobin estimation.** A. W. Coates (*J. Med. Lab. Technol.*, 1954, **12** [1], 47).—The apparatus described allows 12 specimens of blood to be gassed at once and eliminates the continuous discharge of coal-gas into the atmosphere. It consists of a metal cylinder fitted with an air-tight lid and a side valve. Inside is a perforated tube holder. The apparatus can be connected to an automatic shaker. In use, the cylinder containing the tubes of diluted blood is evacuated for 5 min. with a vacuum pump. The cylinder is filled with coal-gas and shaken for 5 to 10 min. Identical results were found for this and the individual gassing method on 200 specimens.

F. W. DIGGINS

1729. **Apparatus for extraction of micro-quantities of lipids.** J. K. Scoggin and O. E. Tauber (*Iowa St. Coll. J. Sci.*, 1953, **28** [2], 165–166).—An extractor (illustrated) consists of a 300-ml Kjeldahl flask fitted with a "cold finger," which allows the condensate to drip into an extraction thimble containing the material under examination. The thimble is fitted into a piece of glass tubing and the whole is easily removed by a glass or metal rod hooked at each end. The apparatus has been used satisfactorily for an investigation of insect lipids, and it has been shown that a 30-min. once-recharged extraction is as efficient as a 12-hr. extraction in a Soxhlet apparatus. N. M. WALLER

1730. **Mounting thin strips for metallographic examination.** A. D. Hopkins (*Metallurgia*, 1954, **49**, 105).—Thin sheets may be held between the coils of a light-tension spring. The pack can then readily be positioned on the base of the die and surrounded by mounting compound in the usual way. G. C. JONES

1731. **A new thickness gauge for textiles.** H. Sommer and F. Winkler (*Faserforsch. u. Textiltech.*, 1954, **5** [2], 77–78).—In the instrument, a dark glass plate with a glass cover plate on to which monochromatic light is directed is attached to the top of the vertically movable loading piston, and the measuring pin of the gauge dial is moved downward until the interference bands begin to move. The zero reading is subtracted from the readings obtained. The apparatus is highly sensitive, thickness at pressures down to 1 g per sq. cm being measurable; the constant load is unaffected by friction. M. TADMAN

1732. **The testing of nicotine filters.** M. Staub and H. Furrer (*Mitt. Lebensm. Hyg., Bern*, 1953, **44** [6], 472–474).—In order to determine the efficiency of cigarette filters, the amount of tar and nicotine in the smoke produced from a given number of cigarettes, with and without the filter, is determined by methods previously described (*Mitt. Lebensm. Hyg., Bern*, 1953, **44**, 371). Because of the high adsorptive power of tobacco for nicotine and tar, the discarded stub of the ordinary cigarette often retains more nicotine and tar than the filter in the filter-tip cigarette. E. HAYES

1733. **Transfusion equipment for medical use.** British Standards Institution (B.S. 2463:1954, 17 pp.).—New standards for equipment used for the taking and administration of blood and for the administration of plasma substitutes and crystalloids are specified. Methods of test of the quality of materials include heating and freezing tests for glass and rubber; detection of inhibitory substances in rubber, and a test of the hydrolytic resistance of the glass. The new specifications are aimed chiefly at standardisation with similar equipment abroad. H. F. W. KIRKPATRICK

1734. **Electron probe techniques for micro-analysis of metallic surfaces.** B.I.S.R.A. Metallurgy (General) Division. New Techniques Committee (*Iron Steel Inst. Rep.*, No. 58, Dec., 1953).—In the Castaing method of local metallographic analysis, a converted electron microscope is used to focus an electron beam on to a metallic specimen. The characteristic X-rays emitted by the elements in the region of the bombarded surface are analysed by a curved crystal quartz X-ray spectrograph and Geiger counter detector. The crystal slide of the spectrometer is calibrated directly in terms of atomic number. Initial microscopic examination permits selection for analysis of an area 2 μ sq. Theoretically all elements of atomic number >11 can be resolved. Measurement of the intensity of characteristic X-rays enables mass concentration of an element to be determined to about 1 per cent. Advantages claimed over light spectrographic analysis include directness of measurement of mass concn., high resolving power, and possibility of obtaining information on lattice type, parameter and orientation. Successful application has included determination of Cu - Zn diffusion curves and analysis of precipitates in alloys.

G. A. BASSETT

Optical

1735. **Techniques of quantitative X-ray spectral analysis. II.** E. E. Vaynshteyn and N. V. Turanskaya (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 346–352).—Methods described previously (*J. Anal. Chem., U.S.S.R.*, 1949, **4**, 323; 1952, **7**, 180; 1952, **7**, 363; 1953, **8**, 311) are developed further and applied to the quant. determination of rare-earth elements. G. S. SMITH

1736. **Air-cooled electrodes for the spectrochemical analysis of powders.** B. J. Stallwood (*J. Opt. Soc. Amer.*, 1954, **44** [2], 171–176).—Some of the irregularities in line intensities that occur when the direct-current arc method is used for the spectrographic analysis of powders can be ascribed to effects of selective volatilisation of the samples or to the effect of the particular matrix on the spectral sensitivity of the elements. These effects can be minimised by cooling the unburned portion of the sample throughout the exposure by an air jet concentric with the sample electrode. This jet also serves to stabilise the arc by surrounding it with a directed current of air. This air-jet method was found to be most effective when the sample craters in the graphite electrodes were made deep relative to their width. B. S. COOPER

1737. **Simple infra-red photometer for chemical analysis.** A. Berton (*Compt. Rend.*, 1954, **238** [4], 477–479).—The photometer, in series with a compensated thermopile and very sensitive galvanometer, makes use of selective emission (H_2O vapour 2·7 μ, CO_2 4·4 μ) from a non-luminous coal-gas flame, the i.r. emission being held constant within 1 per cent. The sample, as a thin solid slice or as liquid or vapour in thin glass cells of thickness up to 50 mm, is placed in the condensed beam from the emitter and acts as selective receiver. The method is used (*i*) to estimate H_2O in CCl_4, C_2Cl_4, Freons, etc., to within 10^{-5} per cent. and in acetone to within 0·05 per cent., as well as CO_2 (0·2 to 5 per cent.) in the atm. to within 5 per cent., (*ii*) to study

the influence of strongly absorbent groups (·OH, :CO, ·CN) on the i.r. absorption of org. compounds and (*iii*) to compare the sp. transparencies of compounds such as benzene, toluene, hexane and *cyclo*hexane. W. J. BAKER

1738. **Apparatus for spectrophotometric study of weak radiations in the near infra-red.** G. Déjardin, J. Janin and M. Peyron (*Compt. Rend.*, 1954, **238** [2], 224–226).—The arrangement described measures relative intensities of weak radiations at 700 to 1100 mμ to within ≃2 per cent. of those obtained by the pyrometric method. Filtered monochromatic radiations from the slit of a rotating-grating spectrometer are made to form an image of the slit on the photo-cathode of an electronic image-converter (*Brit. Abstr. C*, 1951, 474), behind the lighted screen of which is placed a photomultiplier and, for the longer wavelength, a suitable amplifier. The entire receiver is kept in a cool dry atmosphere. Under normal conditions the photomultiplier current is proportional to the flux received by the converter, the spectral sensitivity of the receiver at ≃760 mμ being approx. equal to and at ≃1050 mμ, about 25 times greater than, that of a thermopile receiving the same flux. It is possible to separate clearly the lines in the bands at 750·4 to 751·5, 800·6 to 801·5, 810·4 to 811·5 and 840·8 to 842·5 mμ in the near i.r. spectrum of Al. W. J. BAKER

1739. **A fully automatic recording densitometer for scanning paper-electrophoresis patterns.** D. J. R. Laurence (*J. Sci. Instrum.*, 1954, **31** [4], 137–138).—An automatic recording densitometer having a linear scale is described. Circuit details of the instrument, which employs a photomultiplier operated at constant anode current, are given. G. SKIRROW

Thermal

1740. **Electrical apparatus for measuring the [rapidly fluctuating] temperature [of a fluid in motion].** Ferranti, Ltd. (Inventors: David T. N. Williamson and Donald F. Walker) (Brit. Pat. 706,340, 27.4.51).—Constructional details of an apparatus comprising two high-gain amplifiers in positive feedback association with two bridge networks (each including a temp.-sensitive element in one of its arms) are claimed. J. M. JACOBS

1741. **A rotating combustion bomb for precision calorimetry. Heats of combustion of some sulphur-containing compounds.** W. N. Hubbard, C. Katz and G. Waddington (*J. Phys. Chem.*, 1954, **58** [2], 142–152).—A bomb calorimeter that can be rotated after the combustion is described. The stirring produced by rotation assists the contents of the bomb to reach equilibrium; results for heats of combustion of some S compounds are comparable in precision with those possible for hydrocarbons. Other applications in combustion calorimetry are envisaged, particularly in instances when it is advisable to add a large amount of liquid to the bomb. Standard heats of formation of the following compounds at 25° C are reported: pentane-1-thiol, 3-thiapentane, 2-methylpropane-2-thiol, thia*cyclo*pentane, thia*cyclo*butane and thianthrene. A. JOBLING

Electrical

1742. **Constant-current electro-chemical techniques.** R. N. Adams (*Dissert. Abstr.*, 1954, **14**, 10).—The techniques described comprise: (*i*) the construction of an electronic constant-current supply intended primarily for coulometric titrations, (*ii*) application of a constant-current technique, the derivative polarographic end-point method, to a specific determination, (*iii*) the development of a new polarographic technique, the conventional method being reversed and a current-scanning technique initiated, and (*iv*) a new coulometric titration. R. H. HURST

1743. **Electrochemical apparatus.** Beckman Instruments, Inc., Assees. of Edwin P. Arthur (Brit. Pat. 706,519, Date Appl. 19.11.51, U.S. 23.1.51).—A pressure-equalised reference electrode structure is claimed, which is particularly suitable for the determination of the pH in pressure tanks, jacketed kettles, retorts, etc., while industrial chemical reactions are being carried out in them under pressure. A sheath around the lower portion of the salt-bridge tube provides an annular gas space whereby a minute flow of electrolyte, to renew the liquid junction with the medium being tested, is maintained. J. M. JACOBS

1744. **Electrochemical half-cell.** Beckman Instruments, Inc. (Brit. Pat. 706,549, Date Appl. 19.11.51, U.S. 23.1.51).—A half-cell for use in conjunction with the apparatus described in Brit. Pat. 706,519 (*Anal. Abstr.*, 1954, **1**, 1742) is claimed, *viz.*, a calomel half-cell in which a platinum wire passes through a vol. of electrolyte, superposed on the calomel, to the pool of mercury at the bottom of the closed tube. J. M. JACOBS

1745. **A manual polarograph.** G. O. Jolliffe and C. Morton (*J. Pharm. Pharmacol.*, 1954, **6** [4], 274–280).—A detailed description is given of the assembly and operation of an instrument designed to measure diffusion currents with an accuracy of ±0·1 per cent. To attain this order of accuracy a potentiometric method of determining current is used, the null point being detected by means of an inexpensive type of cathode-ray tuning indicator. The instrument also includes a derivative circuit, which, although tedious in operation in comparison with recording instruments, has the advantage of yielding accurate derivative polarograms. In conjunction with the glass electrode the instrument can also be used for pH measurements. H. F. W. KIRKPATRICK

1746. **Oscillographic polarography.** Ya. P. Gokhshteyn and Yu. A. Surkov (*J. Anal. Chem., U.S.S.R.*, 1953, **8** [6], 323–332).—The construction and use of a new oscillographic polarograph are described, and the effect of various factors on the oscillograms is studied. The experimental results show significant deviations from the results calculated from Randles's equation (*Trans. Faraday Soc.*, 1948, **44**, 327). The method is considerably more sensitive than the normal polarographic method and 10^{-7} *M* solutions can be analysed. G. S. SMITH

1747. **[Mercury electrodes] for polarographic analysis.** Commissariat à l'Énergie Atomique (Brit. Pat. 701,084, 8.5.52, Fr. 10.5.51).—An insulated receptacle (a glass tube) is provided to contain the Hg and to release it at a slow average rate. A metal (Pt, Pt alloy Pd) wire extends from the interior of the tube through a narrow-bore elongation from the walls of which it is spaced to an extent corresponding to a light frictional engagement, thus providing a capillary passage for the flow of the Hg. J. M. JACOBS

See also Abstracts 1445, 1446.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	micro-litre	μl
ampere	amp.	micron	μ
Ångstrom unit	Å	milliampere	mA
anhydrous	anhyd.	milligram	mg
approximate, -ly	approx.	millilitre	ml
aqueous	aq.	millimetre	mm
atmospher-e, -ic	atm.	millivolt	mV
atomic	at.	minimum	min.
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	*M*
calculated	(calc.)	molecul -e, -ar	mol.
calorie (large)	kg-cal.	normal (concentration)	*N*
calorie (small)	g-cal.	number	no.
centimetre	cm	observed	(obs.)
coefficient	coeff.	organic	org.
concentrated	conc.	ounce	oz.
concentration	concn.	part	pt.
constant	const.	patent	pat.
corrected	(corr.)	parts per million	p.p.m.
critical	crit.	per cent. wt. in wt.	per cent. w/w
crystalline	cryst.	per cent. wt. in vol.	per cent. w/v
crystallised	cryst.	per cent. vol. in vol.	per cent. v/v
cubic	cu.	potential difference	p.d.
current density	c.d.	pound	lb
cycles per second	c.p.s.	precipitate	ppt.
decompos -ing, -ition	(decomp.)	precipitated	pptd.
density	ρ	precipitating	pptg.
density, relative	*d* or wt. per ml	precipitation	pptn.
derivative	deriv.	preparation	prep.
dilute	dil.	qualitative, -ly	qual.
direct current	d.c.	quantitative, -ly	quant.
distilled	dist.	recrystallised	recryst.
electromotive force	e.m.f.	refractive index	$n\lambda^t$
electron-volt	eV	relative humidity	R.H.
equivalent	equiv.	revolutions per minute	r.p.m.
experiment, -al	expt.	saponification value	sap. val.
gram	g	saturated calomel electrode	S.C.E.
gram-molecule	mole	second (time)	sec.
half-wave potential	$E_{\frac{1}{2}}$	soluble	sol.
horse-power	h.p.	solution	soln.
hour	hr.	specific gravity	sp. gr.
hydrogen ion concentration	[H·]	specific rotation	$[\alpha]\lambda^t$
hydrogen ion exponent	pH	square centimetre	sq. cm
inch	in.	standard temperature and pressure	s.t.p.
indefinite	indef.	temperature	temp.
infra-red	i.r.	ultra-violet	u.v.
insoluble	insol.	vapour density	v.d.
kilogram	kg	vapour pressure	v.p.
kilovolt	kV	volt	V
kilowatt	kW	volume	vol.
liquid	liq.	watt	W
maxim -um, -a	max.	wavelength	λ
melting-point	m.p.	weight	wt.
microgram	μg		

In addition the following symbols are used—

greater than	>	less than	<
not greater than	≯	not less than	≮
is proportional to	∝	of the order of, approximately	≃

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols: positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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